june 1957

nlgi spokesman

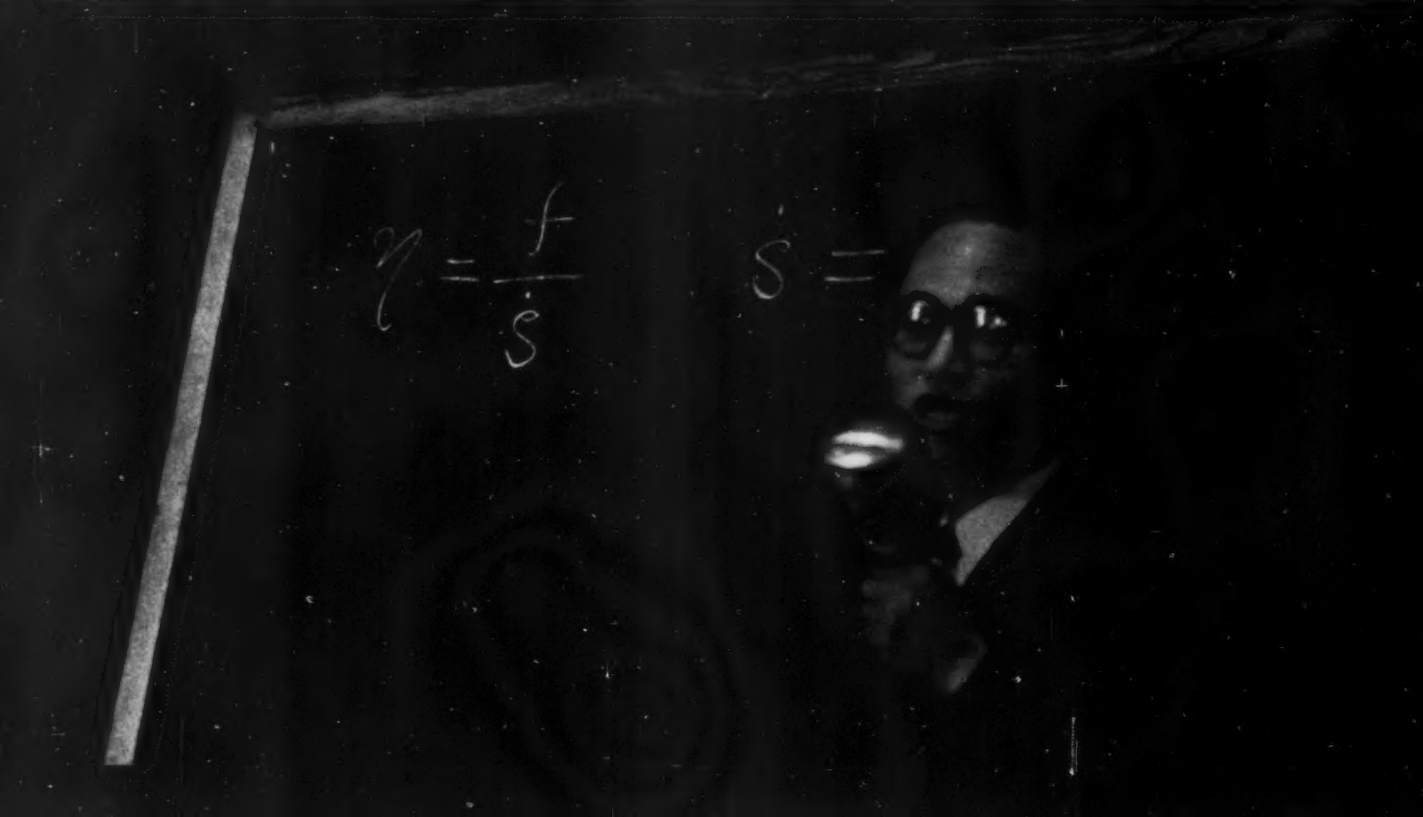
journal of the national lubricating grease institute

A Theory of Thixotropy and its Application to Grease
By S. J. HAHN, T. REE and H. EYRING

NLGI Fellowship Student Stimulating Industry Research
By F. MENSEL

KEL-F No. 90 Grease—A Chemically and Thermally Stable Fluorocarbon Lubricant
By R. J. SEFFL, F. W. WEST and F. J. HONN

Materials, the Key to Aeronautical Progress
By E. M. GLASS



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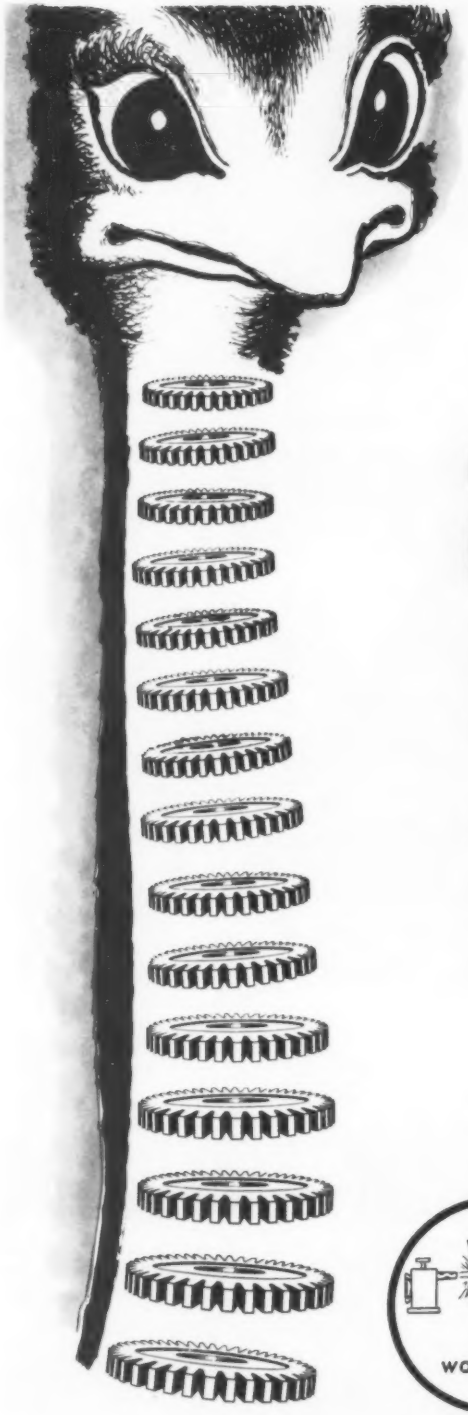

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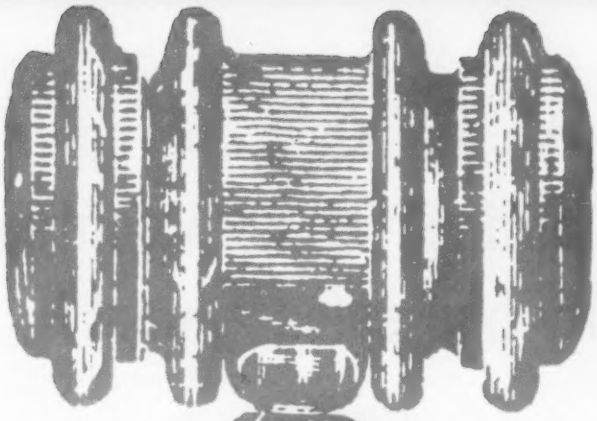

THE PRESIDENT'S PAGE

By J. W. LANE
NLGI
President

QUALITY

"Quality" is a word of different meanings. It can mean among many things an essential property, a distinguishing characteristic, degree of excellence or, even, in a sense often meant to be conveyed, an outstanding or marked excellence.

It is a word much used today, when every manufacturer claims quality for his product with an eye to attracting the consumer's attention. In the manufacture and use of an item such as lubricating grease, quality embraces a variety of concepts. It is not enough to use ingredients which individually are of high quality, but the finished grease must have the quality of suitability for the service or services for which it is recommended. Quality ingredients do not guarantee performance excellence. This can only be proved by extensive testing under actual service conditions.

Tremendous time and effort go into the planning, testing, and making of a lubricating grease which can be offered with assurance to the consumer—in other words, a grease of quality. This is an expensive series of steps, but one that cannot be avoided if the consumer is to be provided with the best possible products.

A quality objective also embraces progressiveness, a desire to be constantly seeking out a better product, even when the present need for it may not be very evident. This is very important in an industry such as ours, since the products are applied to a great variety of equipment which is also usually being constantly improved. This is especially true with respect to the automotive field, where there is a never-ending race to improve equipment and add to the convenience and safety of use, thus throwing added burdens on the lubricant supplier.

We always wince a bit when users talk so confidently about buying lubricants to their individual specifications. There is nothing so conducive to stagnation of progress and a general dropping off in quality. Meeting specification requirements can degenerate into a minimum-quality proposition—and, it is habit-forming and the habit is a bad one. In specification dealings, the supplier is under no obligation to furnish goods better than the specification requirements, since responsibility for performance rests on the purchaser. We wonder if anyone would really be satisfied if this practice were to become widespread.

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Future Meetings

JUNE, 1957

2-7 Society of Automotive Engineers (summer meeting), Chalfonte-Haddon Hall, Atlantic City, N. J.

4-5 Petroleum Packaging Committee, Raddison Hotel, Minneapolis, Minn.

9-14 API Division of Production (midyear committee conference), Muehlebach Hotel, Kansas City.

10-12 Interstate Oil Compact Commission (midyear meeting), Canyon Hotel, Yellowstone National Park.

16-21 ASTM, Chalfonte-Haddon Hall, Atlantic City, N. J.

16-21 ASTM Committee D-2, annual meeting Traymore Hotel, Atlantic City, N. J.

24-25 Michigan Gas Assn. meeting, Grand Hotel, Mackinac Island, Mich.

24-28 American Institute of Electrical Engineers (1957 Summer general meeting), Sharaton-Mt. Royal Hotel, Montreal.

AUGUST, 1957

12-15 National West Coast Meeting, Olympic Hotel, Seattle, Wash.

SEPTEMBER, 1957

9-12 Society of Automotive Engineers, national tractor meeting and production forum, Hotel Schroeder, Milwaukee.

10-11 Petroleum Packaging Committee, Point Edward, Ontario, Canada.

11-13 National Petroleum Association (55th annual meeting), Traymore Hotel, Atlantic City, N. J.

22-24 Tenth Annual Meeting of the Independent Oil Compounders Association. Carter Hotel, Cleveland, Ohio.

30-Oct. 2 American Oil Chemists' Society (1957 Fall meeting), Netherland Plaza Hotel, Cincinnati.

OCTOBER, 1957

1-2 Texas Mid-Continent Oil & Gas Assn. (38th annual meeting), Texas Hotel, Fort Worth.

1-5 National Aeronautic Meeting, Aircraft Production Forum, and Aircraft Engineering Display, The Ambassador, Los Angeles, Calif.

6-10 ASTM Committee D-2 meeting, Sheraton-Park Hotel, Washington, D. C.

7-11 American Institute of Electrical Engineers (1957 Fall general meeting), Morrison Hotel, Chicago.

9-12 National Tractor Meeting and Production Forum, Hotel Schroeder, Milwaukee, Wis.

13-16 API Division of Refining (midyear meeting), Penn-Sheraton Hotel, Philadelphia.

16-20 API Marketing Div. Marketing Research Committee, Camelback Inn, Phoenix.

28-30 NLGI Annual Meeting, Edgewater Beach Hotel, Chicago, Ill.

NOVEMBER, 1957

4-6 National Transportation Meeting, Hotel Statler, Cleveland, Ohio.

5-6 National Diesel Engine Meeting, Hotel Statler, Cleveland, Ohio.

6-8 National Fuels and Lubricants Meeting, Hotel Statler, Cleveland, Ohio.

11-14 API 37th Annual Meeting, Conrad Hilton and Palmer House, Chicago, Ill.

MARCH, 1958

4-6 Passenger Car, Body and Materials Meeting, Sheraton-Cadillac, Detroit, Michigan.

31-Apr. 2 National Production Meeting and Forum, The Drake, Chicago, Ill.

APRIL, 1958

16-18 National Petroleum Association, Cleveland, Ohio

JUNE, 1958

8-13 Summer Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

22-28 ASTM 61st Annual Meeting Hotel Statler, Boston, Mass.

SEPTEMBER, 1958

10-12 National Petroleum Association, Atlantic City, N. J.

OCTOBER, 1958

20-22 National Transportation Meeting, Lord Baltimore Hotel, Baltimore, Md.

22-24 National Diesel Engine Meeting, Lord Baltimore Hotel, Baltimore, Md.

27-29 NLGI ANNUAL MEETING, Edgewater Beach Hotel, Chicago, Ill.

NOVEMBER, 1958

5-6 National Fuels and Lubricants Meeting, The Mayo, Tulsa, Okla.

***MARCH, 1959**

3-5 Passenger Car, Body, and Materials Meeting, Sheraton-Cadillac, Detroit, Michigan.

JUNE, 1959

14-19 Summer Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

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THE COVER

Author Sang Joon Hahn was photographed while delivering "A Theory of Thixotropy and its Application to Grease" at the NLGI Annual Meeting in Chicago. Co-authors of the article are Taikyue Ree and Henry Eyring, all of the University of Utah. Hahn holds the NLGI fellowship for 1956-1957 under the guidance of Dr. Eyring who is dean of the Utah University graduate school. Before joining forces at Utah the authors had worked together in Japan and this country. Dr. Ree was professor of chemistry at Kyoto where he met Hahn, and later Ree studied with Eyring at Princeton University. Article begins on page 12.

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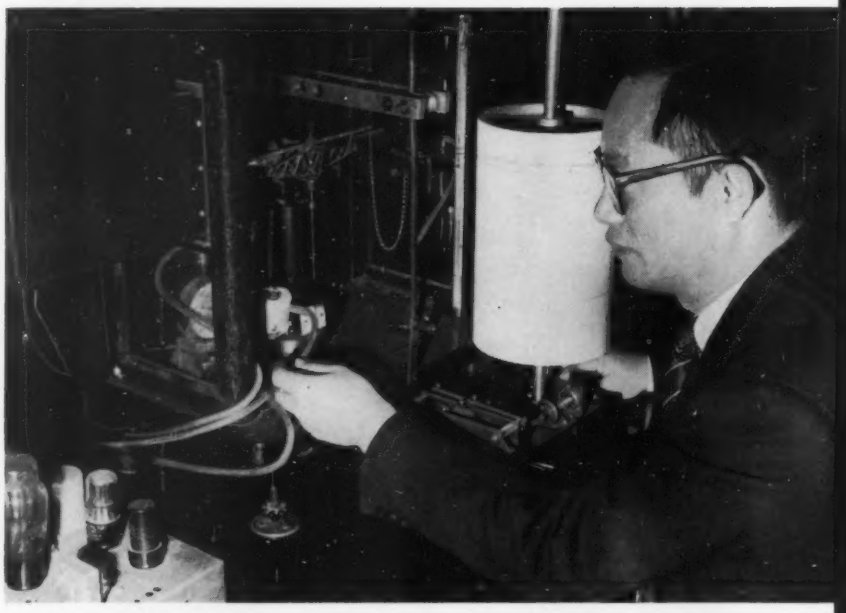

SANG JOON HAHN, a co-author and NLGI Fellowship recipient at work.

and its Application to Grease

By S. J. Hahn
T. Ree and
H. Eyring
University of Utah

Introduction

GREASE IS A mixture composed of soap and lubricating oil. According to recent studies,[1,2] a grease is considered as a three-dimensional network of soap fibers with the lubricating oil trapped between fibers. The network yields under an applied force, and thus the grease flows. However, the original structure is restored when the applied force is removed, that is, grease has a thixotropic nature.

In addition to grease, many thixotropic systems have been found.[3,4,5] No theory, however, has been proposed which describes thixotropy correctly and quantitatively. We study here the rheological systems which have the following properties: (1) while flowing, the system undergoes a structural change which is a function of shear rate only; (2) the structural change is a function of shear rate as well as of time. The flow equation in case (1) is applied in explaining "the Ostwald flow curves," whereas the equation in case (2) is used to describe the thixotropy of grease.

ABSTRACT

We assume that there are two kinds of flow units in a flow system, i.e., non-Newtonian (entangled) and Newtonian (disentangled) units. The non-Newtonian units transform into the Newtonian by a stress. Two cases exist: (1) the transformation is in a kinetic equilibrium; (2) it is irreversible. In case (1), the concentrations of flow units are independent of time, while the concentrations in case (2) are time-dependent. The idea of the transformation is introduced into the Ree-Eyring theory of non-Newtonian flow, and two flow equations corresponding to case (1) and (2) are obtained. "The Ostwald flow curve" is explained by the flow equation for case (1), whereas "the thixotropy curve" is well described by the equation for case (2). The theoretical flow equations are applied to the experimental flow curves of the following systems: rubber dissolved in toluene, nitrated American raw cotton dissolved in butyl acetate, nitrated pine pulp in butyl acetate, melted sulfur, gelatine sol, melted cholesteryl butylate, and Grease S. Except for Grease S, which is thixotropic, all the systems show good Ostwald curves. The agreement between theory and experiment is satisfactory.

Flow Accompanying the Structural Change Which Is Time-Independent

According to the Ree-Eyring theory of non-Newtonian flow,[6] viscosity is given by the following equation:

$$\eta = \sum_{n=0}^{\nu} \frac{x_n \beta_n}{\alpha_n} \frac{\sinh^{-1} \beta_n \dot{s}}{\beta_n \dot{s}} \tag{1}$$

where,
$$\alpha = \lambda\lambda_2\lambda_3/2kT \tag{2}$$

and
$$\beta = \left(\frac{\lambda}{\lambda_1} 2k'\right)^{-1} \tag{3}$$

λ, λ_1, λ_2, λ_3, and k' are the customary parameters in Eyring's theory of flow.[7] β is called the relaxation time since it has the dimensions of time, $\dot{s}$ is the rate of shear. The subscript, n, signifies that the attached quantities belong to the nth kind of flow units, the zeroth kind being ascribed to solvent flow units. There are $\nu + 1$ kinds of flow units in the system concerned and x_n is the fractional area occupied by the nth kind of flow units. Introducing the relation $f = \eta\dot{s}$, into (1), one obtains

$$f = \sum_{n=0}^{\nu} \frac{x_n}{\alpha_n} \sinh^{-1} \beta_n \dot{s} \tag{4}$$

Here f is stress (shear stress per unit area). If there is one kind of Newtonian flow unit and one kind of non-Newtonian unit for a solute, equation (4) is transformed into

$$f = \frac{x_1\beta_1}{\alpha_1} \dot{s} + \frac{x_2}{\alpha_2} \sinh^{-1} \beta_2 \dot{s} + \frac{x_0\beta_0}{x_0} \dot{s} \tag{5}$$

Here, subscripts 1 and 2 indicate the Newtonian and non-Newtonian Solute units respectively, and solvent units have been treated as Newtonian units. Equation (5) is essentially the same formula developed by Powell and Eyring for lime base grease.[8a]

It is appropriate to consider here the structure of the Newtonian and the non-Newtonian flow units. In polymeric systems, intermolecular entanglement will determine the relaxation times of flow units. Thus, disentangled (folded) units serve as Newtonian units, whereas entangled (unfolded) units as non-Newtonian. (β's for non-Newtonian units in a system are larger than β's for Newtonian units.) The state of entanglement will not be changed by low stresses. High stresses, on the contrary, tend to break bonds which may not re-form immediately. Thus, entangled (non-

Newtonian) units transform into disentangled (Newtonian) units.

We consider the case where entangled and disentangled units are in kinetic equilibrium, and the system shifts toward disentanglements with increasing stress. The free energy curve for the transition is represented by Figure 1, where E and D indicate the entangled and the disentangled units, respectively. The transition, E $\longrightarrow$ D, is promoted by elastic energy stored up in the flow unit. Such a unit is formally represented as a Maxwell unit—a spring in series with a dashpot. The elastic energy, w, stored up during the relaxation time, β_2, is given by

$$w = \int_0^{s_1} f\,ds = G(\beta_2 \dot{s})^2/2 = c\dot{s}^2 \qquad (6)$$

Here, G is the spring constant of the unit, and the relation, $f = Gs$ and $s_1 = \beta\dot{s}$, have been substituted; s_1 is the strain at the time of relaxation.

Let C_e and C_d be the mole fractions of the entangled and disentangled units, respectively, in a shear surface at time t. Then, the net rate of transition, E $\longrightarrow$ D, is given by

$$-\frac{d\,C_e}{dt} = C_e k_f e^{\frac{\mu c\dot{s}^2}{kT}} - C_d k_b e^{\frac{(1-\mu)c\dot{s}^2}{kT}} \qquad (7)$$

For an unsymmetrical barrier, μ will be different from one-half; k_f and k_b are specific rates for the forward and backward reactions at zero stress. The condition of steady-flow is that the left side of (7) is zero. Thus, we obtain

$$C_e = 1/(1 + Ke^{\frac{c\dot{s}^2}{kT}}) \qquad (8a)$$

$$C_d = Ke^{\frac{c\dot{s}^2}{kT}}/(1 + Ke^{\frac{c\dot{s}^2}{kT}}) \qquad (8b)$$

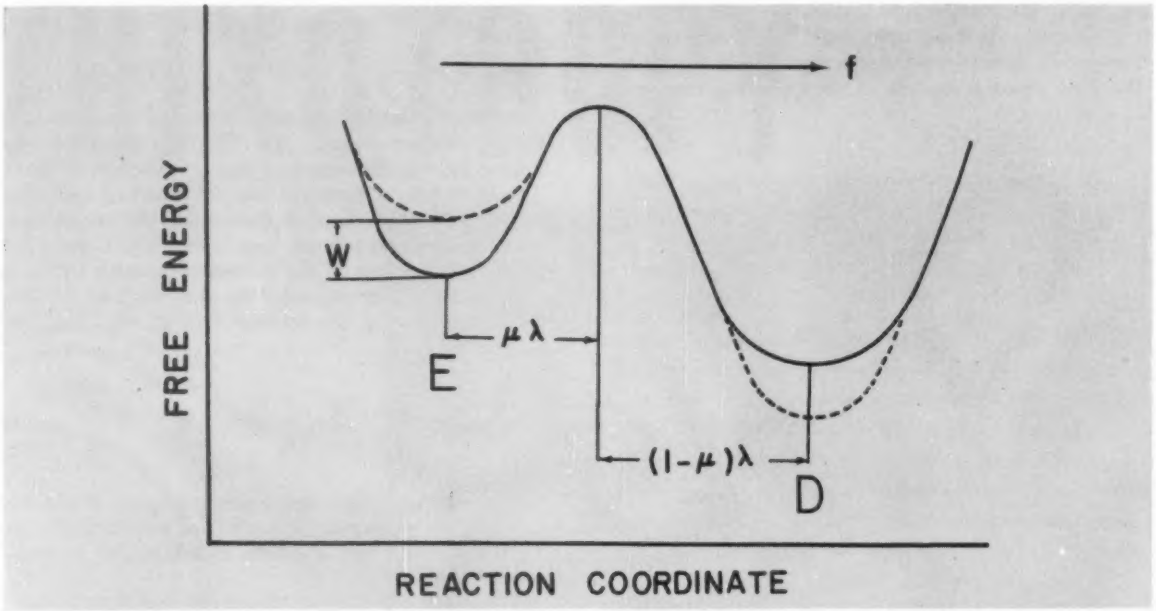

FIGURE 1 The free energy curve for transition, entanglement-disentanglement. E and D are entangled and disentangled.

Where $K = k_f/k_b$ (the equilibrium constant at zero stress), and the relation, $C_e + C_d = 1$, has been introduced. x_1 and x_2 are related to C_d and C_e by the following equations:

$$x_1 = N_s C_d A_d \qquad (9a)$$

$$x_2 = N_s C_e A_e \qquad (9b)$$

where N_s is the total number of solute flow units per unit area, and A_d and A_e are the areas occupied by one of the disentangled and the entangled units, respectively.

Generally, the structural change does not accompany volume change; thus we may assume $A_d = A_e$. The normalization condition for fractional areas for molten

polymers and for polymer solutions are,

$$x_1 + x_2 = 1 \qquad (9c)$$

$$\text{and} \quad x_1 + x_2 + x_0 = 1 \quad \text{respectively} \qquad (9d)$$

From (9a)–(9c), one obtains $N_sA_1 = N_sA_2 = 1$ for molten polymers, i.e., $x_1 = C_d$ and $x_2 = C_e$. Substitution of equations (8) and (9) into (5) yields

$$f' = \frac{N_sA_1}{1+Ke^{\frac{c\dot{s}^2}{kT}}} \left(\frac{\beta_1}{\alpha_1} Ke^{\frac{c\dot{s}^2}{kT}} \dot{s} + \frac{1}{\alpha_2} \sinh^{-1} \beta_2 \dot{s} \right) \qquad (10)$$

where $f' = f$ and $N_sA_1 = 1$ for molten polymers, and $f' = f - \eta_0\dot{s}$ for *dilute* solutions, η_0 being the solvent viscosity, and $x_0 \simeq 1$. If $c = O$ (i.e., $G = O$ from (6)), equation (10) reduces to (5). That is, if the unit does not have elasticity, the aforementioned structural change does not occur.

Flow Accompanying the Structural Change Which Is Time-Dependent

Next, we consider the case where there is no kinetic equilibrium in the transition, $E \longrightarrow D$. In this case, the left-hand side of (7) is not zero. Equation (7) is well approximated by the following equation:

$$-\frac{d\,C_e}{dt} \simeq C_e k_f e^{\frac{\mu c\dot{s}^2}{kT}} \qquad (11)$$

Experimentally, $\dot{s}$ is changed with time according to the relation:

$$\dot{s} = \omega t \qquad (12)$$

Here, ω is a constant which is obtained from the experimental conditions. For example, if $\dot{s}$ is raised from zero to $\dot{s}_m$ in time t_m, then $\omega = \dot{s}_m/t_m$. Integrating (11), into which (12) was substituted, yields:

$$C_e = (C_e)_0 e^{-k_f\gamma I} \qquad (13)$$

where, $(C_e)_0$ is C_e at $t = 0$,

$$I = \int_0^y e^{y^2} dy \qquad (14)$$

$$\text{and} \quad y = t/\gamma = t/\left(\frac{kT}{\mu C\omega_2}\right)^{1/2} = \dot{s}/\left(\frac{kT}{\mu C}\right)^{1/2} \qquad (15)$$

Introducing (9) and (13) into (5), one obtains

$$f = (1-(C_2)_0 e^{-k_f\gamma I}) \frac{1}{\alpha_1} \beta_1 \dot{s} + \frac{1}{\alpha_2} (C_2)_0 e^{-k_f\gamma I} \sinh^{-1} \beta_2 \dot{s} \qquad (16a)$$

Here, the system has been treated as a molten polymeric type since usually thixotropy occurs in concentrated solutions. Equation (16a) is approximated by the following equation:

$$f = [1-(C_2)_0(1-k_f\gamma I)] \frac{1}{\alpha_1} \beta_1 \dot{s} + \frac{1}{\alpha_2} (C_2)_0 (1-k_f\gamma I) \sinh^{-1} \beta_2 \dot{s} \qquad (16b)$$

According to (16a) and (16b) the flow curve, $\dot{s}$ versus f, is time-dependent. That is, the curve depends on how fast $\dot{s}$ is raised to $\dot{s}_m$, since (16) includes γ which is a function of ω. Here, $\dot{s}_m$ is a fixed maximal shear rate in a series of experiments.

Determination of Parameters

The Ostwald Curve. Figure 2(B) exemplifies the Ostwald curve. It has the following characteristics: (1) there is an inflection point in the linear (nonlogarithmic) plot of $\dot{s}$ versus f; (2) the part of the flow curve beyond the inflection point is extrapolated to the origin (cf. Figure 2(B) and also 2(A)). The nature of the Ostwald curve is explained by the assumptions: (1) there are at least two kinds of flow units–Newtonian and non-Newtonian; (2) the non-Newtonian (entangled) units transform into the Newtonian (disentangled) units by stresses, eventually being completely changed to the Newtonian units by high stresses (shear rates); (3) the transformation is in kinetic equilibrium, i.e., the concentrations of the two kinds of flow units are time-independent. Using these assumptions, equation (10) has been derived. Thus it describes the properties of the Ostwald curve correctly. That is, (10) transforms into the following Newtonian equation at high $\dot{s}$:

$$f' = \frac{N_sA_1\beta_1}{\alpha_1} \dot{s} \qquad (17)$$

whereas (10) may be written as (5) at very low $\dot{s}$, with the following equalities:

$$(x_1)_0 = N_sA_1K/(1 + K) \qquad (18a)$$

and

$$(x_2)_0 = N_sA_1/(1 + K) \qquad (18b)$$

where, $(x_1)_0$ and $(x_2)_0$ are the corresponding quantities as $\dot{s} \to 0$. From the properties of equation (10) mentioned above, the parameters of (10) are readily determined. The procedure is as follows: (1) Extend the upper part of the Ostwald curve beyond the inflection point toward the origin. The slope of the extended straight line gives $\frac{N_sA_1\beta_1}{\alpha_1}$ (cf. equation

(17)). (2) Apply (5) to the low range of $\dot{s}$ of the flow curve, $(x_1)_0\,\beta_1/\alpha_1$, $(x_2)_0/\alpha_2$ and β_2 are determined by the Ree-Eyring method.[6] From the results of these two procedures, $\frac{N_s A_1 \beta_1}{\alpha_1}$, K, $\frac{N_s A_2}{\alpha_2}$, and β_2 are evaluated. (3) Apply equation (10) to the curve in the neighborhood of the inflection point. c is calculated, since it is the only parameter which is unknown in (10). By repeating the above procedures several times, the best values of all the parameters of (10) are obtained.

Thixotropy Curve. A thixotropy curve is composed of two parts: an "up curve" and a "down curve" (cf. Figure 3). The up curve is the flow curve which is obtained by measuring $\dot{s}$ with increasing f. The flow curve in the reverse direction is the down curve. As shown in Figure 3, a thixotropy curve has a hysterisis loop. This fact is explained as follows: In the up curve, non-Newtonian units transform to Newtonian units as in the Ostwald curve. The only difference from the Ostwald curve is that in the up curve there is no kinetic equilibrium for the transformation, i.e., the transformation is irreversible. Only with prolonged elapsed time, the transformed units return to the original non-Newtonian state. Thus, in the down curve the concentrations of Newtonian and non-Newtonian units are constant and are determined by the concentration at the apex of the up curve. Consequently, the down curve does not coincide with the up curve, because the system for the down curve is necessarily more fluid.

The down curve is well expressed by equation (5).[6, 8a] By applying (5) with $x_0 \simeq 0$ to the down curve, the parameters, $(x_1)_m\beta_1/\alpha_1$, $(x_2)_m/\alpha_2$, and β_2 are obtained. This β_2 is used for the calculation of (16b). The subscript, $_m$, indicates that the attached quantities are functions of the *maximal* (apex) point of the up curve. The determination of the parameters of (16b) involves the following procedures: (1) Apply (5) with $x_0 = 0$ to the up curve in the range of small $\dot{s}$, and determine the parameters, $(x_1)_0\beta_1/\alpha_1$, $(x_2)_0/\alpha_2$, and β_2. This β_2 agrees well with β_2 determined from the down curve. (2) Assume $x_1\beta_1/\alpha_1$ does not change appreciably, while x_2/α_2 does change in the range of small $\dot{s}$'s only slightly larger than those in procedure (1). Thus, the flow equation in this range of $\dot{s}$ is given by

$$f = \frac{(x_1)_0\beta_1}{\alpha_1}\,\dot{s} + \frac{1}{\alpha_2}\,(C_2)_0(1-k_f\gamma I)\sinh^{-1}\beta_2\dot{s} \qquad (19)$$

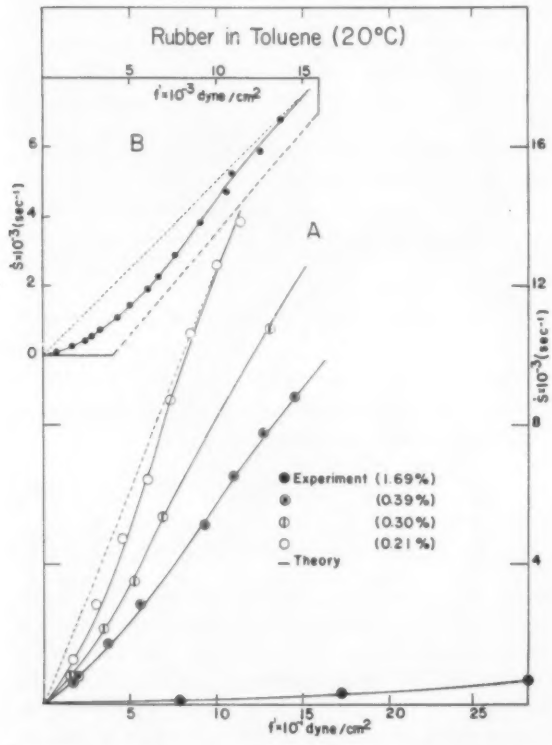

FIGURE 2 Flow curves of rubber dissolved in toluene at 20°C. Figure 2 (B) exemplifies the Ostwald curve.

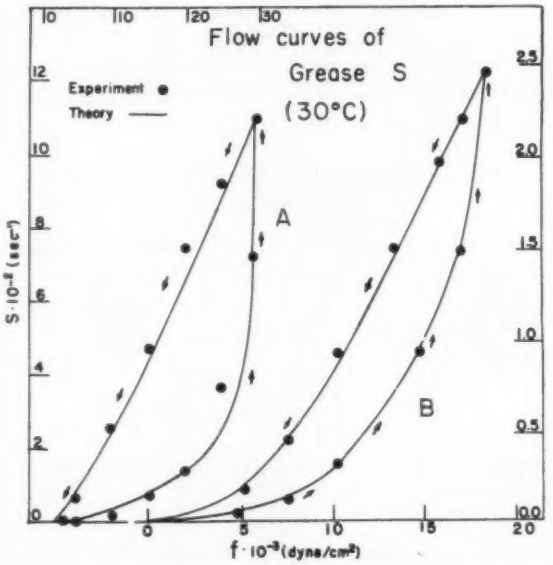

FIGURE 3 Gives thixotropy curves of grease S at 30°C. The left and upper scales are referred to curve A, and the right and lower scales are referred to curve B.

Calculate $f-[(x_1)_0\beta_1/\alpha_1]\,\dot{s}$ and $[(x_2)_0/\alpha_2]\sinh^{-1}\beta_2\dot{s}$, and make the following ratio:

$$\frac{f-[(x_1)_0\beta_1/\alpha_1]\,\dot{s}}{[(x_2)_0/\alpha_2]\sinh^{-1}\beta_2\dot{s}} = 1-k_f\gamma I \simeq 1-k_f\frac{\dot{s}}{\omega} \qquad (20)$$

Equation (20) follows from (19). The approximate equality of (20) is obtained by introducing $I \simeq y = \dot{s}/(\gamma\omega)$. (3) Plot the left-hand side of (20) against $\dot{s}$. Then a straight line is obtained, the slope of which is $-k_f/\omega$. Thus k_f is calculated, ω being known. (4) At the apex, we have the relation:

$$\frac{(x_2)_0}{\alpha_2}(1-k_f\gamma I_m) = \frac{(x_2)_m}{\alpha_2} \qquad (21)$$

Thus, the value of γI_m is calculated, the parameters, $(x_2)_0/\alpha_2$, k_f, and $(x_2)_m/\alpha_2$ being known. (5) Calculate the following ratio:

$$\frac{\omega\gamma I_m}{(\dot{s})_m} = \frac{\omega\gamma I_m}{\omega\gamma y_m} = \frac{I_m}{y_m} \qquad (22)$$

Here (15) has been used. Make a graph of I versus y, where the values of I are obtained by a graphical method. Draw a straight line with a slope of I_m/y_m given by (22) and passing through the origin. The crossing point of the straight line with the curve of I versus y gives I_m and y_m. Then the factors, γ and $(kT/\mu c)^{1/2}$, are calculated from (15), t_m being known, and consequently the values of I at any desired values of $\dot{s}$ are read from the curve of I versus y. (6) Calculate the second term, II, on the right of (16b) using the values of parameters found, and calculate the ratio $(f-II)/\dot{s}$, using the up curve. (7) Plot $(f - II)/\dot{s}$ against $k_f\gamma I$. The slope of the plot (a straight line) yields $(C_2)_0\beta_1/\alpha_1$, while the intercept with the ordinate axis gives $(C_1)_0\beta_1/\alpha_1$. Thus, $(C_2)_0$ is calculated.

Application

Examples of Ostwald Curves. The flow curves in Figure 2 are those for the toluene solutions of natural rubber in various concentrations. By applying (10) to the experimental points which are due to Reiner and Reiner,[8b] the parameters listed in Table I were obtained. The theoretical curves are calculated from equation (10) using these parameters. The agreement with experiment is satisfactory. Philippoff[9] repeated the same experiment with the Reiners', extending the range of $\dot{s}$ and f more than the Reiners had done. We plotted Philippoff's results using linear scales for $\dot{s}$ and f, and found that his data show good Ostwald curves.

The flow data of nitrated American raw cotton dissolved in butyl acetate[10] and of nitrated pine pulp in butyl acetate[10] are shown in Figures 4 and 5, respectively. The parameters for these cases are tabulated in Table I, and the theoretical curves are calculated from (10) using these parameters. One may see that equation (10) describes the flow curves of these cases correctly. As in Figure 2, the nature of the Ostwald curve is more distinct in concentrated solutions than in dilute solutions.

In Figure 6, the flow data of gelatine sol in water,[11] melted cholesteryl butylate,[12] and melted sulfur[13] are represented. The theoretical curves are calculated from (10) by using the parameters in Table I. Also in these cases, the Ostwald curves are very well expressed by (10).

Thixotropy in Grease. Figure 3 represents the hysteresis curves for Grease S.[14] The experimental points in A were obtained with $\dot{s}_m = 1100\ sec^{-1}$, and $t_m = 30$ sec., and those in B were obtained with $\dot{s}_m = 250\ sec^{-1}$ and $t_m = 30$ sec. That is, the values of

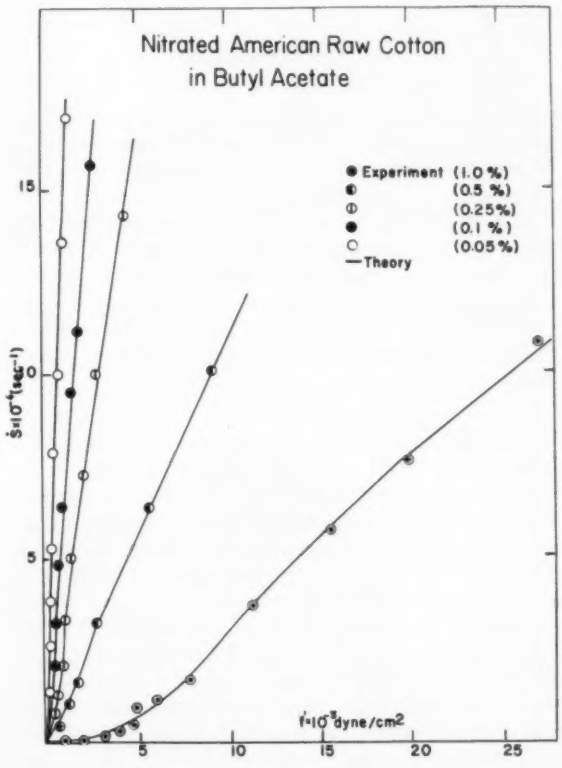

FIGURE 4 Flow curves of nitrated American raw cotton dissolved in butyl acetate. The parameters for these cases are tabulated in Table 1 which appears on page 19.

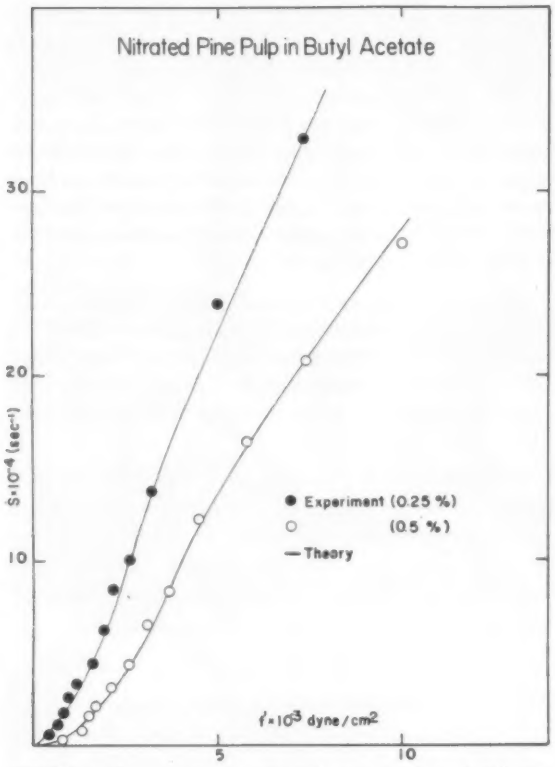

FIGURE 5 Flow curves of nitrated pine pulp in butyl acetate. The parameters are tabulated in Table I on page 19 and the theoretical curves are calculated from (10).

ω in A and B are 36.7 and 8.33 sec.$^{-2}$, respectively. By applying (16b) to the experimental data in A and B, we obtained the parameters listed in Table II. The theoretical curves of A and B were calculated from (16b) using the parameters in Table II. One sees that the agreement between theory and experiment is satisfactory.

Discussion

Non-Ostwald Curves. There are many non-Ostwald curves which have been treated, however, as the Ostwald curves in the literature.[9b,15] That is the flow systems for these non-Ostwald curves have been considered as accompanying structural change by stress. All of the flow data, however, are reported in the double logarithmic plots of $\dot{s}$ versus f. The shapes of the logarithmic curves are very similar to the Ostwald curve in Figures 2, 4, 5, and 6. We replotted the logarithmic curves using a linear scale of $\dot{s}$ versus f, and found that the curves fall into two classes. One is the true Ostwald curve, i.e., it is represented by equation (10), while the other is expressed by (5), i.e., it does not involve a structural change. The details, however, will not be given here. We treat here only the true Ostwald curves.

Examination of the Flow Parameters. The data in Tables I and II reveal several very interesting facts. For a given fluid, the value of c/kT in Table I is about constant irrespective of concentration; it does change somewhat from fluid to fluid for dilute solutions, but the order of magnitude is constant regardless of the concentration and of the fluid used. Other parameters in Table I change with concentration. Table II shows that $(C_2)_0$ is 75%, but it decreases to 28% when $\dot{s}$ increases from zero to 1100 sec^{-1} in 30 sec, while it decreases to 58% when $\dot{s}$ increases from zero to 250 sec^{-1} in 30 sec. One sees that non-Newtonian units transform to Newtonian more rapidly at high $\dot{s}$ than at low $\dot{s}$. The detailed discussions on the parameters in Tables I and II and a fuller presentation of thixotropic theory will be given elsewhere.

Polymers Used as the Lubricating-Oil Additives. It is a common practice to blend high polymers with greases in order to improve the properties of the lat-

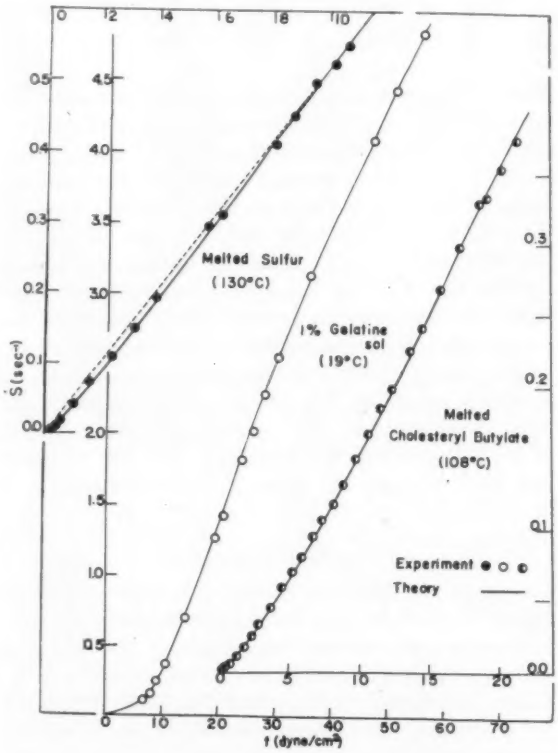

FIGURE 6 Flow curves of melted sulfur (130°C), 1% gelatine sol (19°C), and melted cholesteryl butylate (108°C). Outside left and upper scales are referred to melted sulfur; inside left and outside lower scales are referred to 1% gelatine sol; and the right and inside lower scales are referred to melted cholesteryl butylate.

ter. For example, high polymers are used as "the pour-point depressants" and as "the viscosity-index (V.I.) improvers."[16,17] Thixotrophy is a non-desirable property of a grease in some cases.[18] For example, a thixotropic grease requires a high torque for starting a machine, while low power is needed for keeping it running; it is apt to lead to difficulties in pumping through dispensing lines or to actual "freezing" of the system; and a tight bearing housing is required for preventing leakage of the broken down grease. Thus, non-thixotropic grease is desirable, i.e., the grease giving the Ostwald flow curve is better than the one with the thixotropy curve. We believe certain high polymers will also act as inhibitors for thixotropy when used as a lubricating-oil additive.

The mechanism of V.I. improver action is not completely understood, but present concepts explain the action as follows[19,20]: At low temperatures, the polymeric additive is only slightly soluble, and is solvated to a small extent. As temperature rises, more and more of the polymer goes into solution, and the high viscosity of the polymer contributes to a greater extent toward the total viscosity of the lubricant blend. In addition to this kind of action, we believe, the polymer re-enforces the network of soap fibers. The re-enforcement will be greater at high temperatures than at low ones, since at high temperatures the polymer molecules uncoil and become entangled. Thus, the

TABLE I

Values of the Parameters for Ostwald Curves

	Temp. °C	*Volume Conc.* (%)	K	$N_s A_1/\alpha_2$ *dyn./cm*$_2$	β_2 *Sec.*	$N_s A_1 \beta_1/\alpha_1$ *dyn.sec./cm*^{2}	c/kT *Sec.*^{2}	η_0
		1.69	9.0	690.0	0.01	0.2004	2.5×10^{-9}	0.0073
Rubber in	20	0.39	10.0	54.0	0.003	0.0166	4.5×10^{-9}	
Toluene[8]		0.30	10.0	52.0	0.002	0.0124	2.0×10^{-9}	
		0.21	10.0	51.0	0.001	0.0081	4.0×10^{-9}	
Nitrated		1.00	6.0	1888.0	30.0	0.2667	0.50×10^{-9}	0.0059
American		0.50	11.0	900.0	3.6	0.0919	0.80×10^{-9}	
raw cotton		0.25	25.0	790.0	0.2	0.0287	0.33×10^{-9}	
in Butyl	20*	0.10	30.0	500.0	0.04	0.0157	0.51×10^{-9}	
acetate[10]		0.05	32.0	365.0	0.006	0.0081	0.80×10^{-9}	
Nitrated								
Pine pulp		0.50	8.0	1820.0	0.01	0.0357	0.10×10^{-9}	0.0059
in Butyl	20*	0.25	9.0	1000.0	0.003	0.0237	0.26×10^{-9}	
acetate[10]								
Gelatin								
sol in	19	1.00	9.0	11.5	500.0	11.415	0.3	0.0103
water[11]								
Melted								
Cholesteryl	108		9.0	4.4	500.0	58.3	6.0	
butylate[12]								
Melted								
Sulfur[13]	130		6.0	0.6	2000.0	19.5	10.0	

*It has been estimated from the solvent viscosity, η_0, since the author has not specified the temperature.

TABLE II

Values of the Parameters for Thixotropy Curves of Grease S*

	Temp. °C	$\sqrt{\frac{kT}{\mu c \omega^2}}$ sec.	k_f sec.$^{-1}$	β_1/α_1 dyn. sec./cm^2	β_2 sec.	$1/\alpha_2$ dynes/cm$_2$	$(C_2)_0$	$(C_2)_m$	$\omega_{calc.}$
$\dot{s}$ 1100 sec.$^{-1}$ in 30 seconds	30	21	0.003	26	3	4000	0.75	0.278	31.7
$\dot{s}$ 250 sec.$^{-1}$ in 30 seconds	30	21	0.003	120	3	2000	0.75	0.580	8.2

*The parameters were obtained from Ref. (14).

polymer additive reduces the thixotropic character of the grease, and consequently, increases the viscosity index.

Acknowledgments

We wish to thank Dr. N. Hirai for valuable discussions and kind advice. One of us (S. J. H.) expresses his appreciation to the National Lubricating Grease Institute for the fellowship given to him.

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About the Authors

SANG JOON HAHN *received the National Lubricating Grease Institute fellowship for* 1956-1957 *and is studying toward a doctorate degree in physical chemistry at the University of Utah. Sang is working under the supervision of Dr. Henry Eyring and is principally concerned with the mechanisms of liquid flow. A graduate of the Seoul National University in* 1948, *Hahn instructed in chemistry at the Ewha University in Korea from* 1948 *to* 1955, *then began graduate work at Utah University.*

DR. TAIKYUE REE *has been a research professor in chemistry at the University of Utah since* 1949. *He received B. S. and Sc. D. degrees in chemistry from Kyoto Imperial University, Japan in* 1931. *He became a professor of chemistry there following graduation and achieved professorship in* 1943. *In* 1945 *he was named dean and professor of the college of liberal arts and sciences at the Seoul National University, Korea, where he remained until taking his present position at the University of Utah. Dr. Ree has devoted his time to the study of chemical kinetics, catalysis, reactivity of organic substances, rheology, quantum chemistry.*

DR. HENRY EYRING *has been dean of the graduate school, University of Utah since* 1946. *He is also associate editor of "Textile Research Journal." From* 1944 *to* 1946 *he was research director of the Textile Foundation, worked with the Office Science Research & Development, U. S. Navy in* 1944 *and received the Alumni Achievement Award from Arizona University in* 1947. *Dr. Eyring received the degrees of B. S. and M. S. from Arizona University, Ph. D. in chemistry from California, and Honorary Science Degree from Utah in* 1952, *Northwestern,* 1953 *and Princeton in* 1956. *He is a member of the National Academy, Soc. Rheol., winning the Bingham Medal* 1949, *Chem. Soc., winning the Nichols Medal in* 1951, *and Philos. Soc.*

NLGI Fellowship Student Stimulating Industry Research at Utah University

By FRANK MENSEL, University of Utah

Few people have traveled farther or sacrificed more for an education than Sang Joon Hahn. Sang holds the National Lubricating Grease Instutitute fellowship for 1956-57.

Getting into graduate work in an American university—he is studying toward a doctorate in physical chemistry at the University of Utah in Salt Lake City—meant a trip almost half way around the world to a country and a campus he had never seen. His home is Seoul, South Korea.

It meant, too, the personal sacrifice of a long separation from his family. When he started out for Salt Lake City in late summer, 1955, he left Mrs. Hahn and their three children in Seoul.

"Probably I won't see them for at least three years," he said. At the earliest, he will complete his graduate program sometime in late 1958, he said.

Studies Mechanisms of Liquid Flow

Sang, 35, is devoting his graduate research to studies on the mechanisms of liquid flow. He holds the only fellowship given by the National Lubricating Grease Institute, a $2,500 grant. The Institute established its fellowship at the University of Utah because the Institute of Rate Processes makes the Salt Lake campus one of the foremost centers of the world in the study of flow mechanics and other reaction processes.

Sang's work is supervised by Dr. Henry Eyring, one of America's foremost authorities on rheology—a relatively new science devoted to the mechanics of flow. (The term "rheology" is formed from a Greek word which means "flow.") In his research Sang also has the guided help of a man he knew years ago at the University of Kyoto in Japan, Dr. Alexius Taikyue Ree. Dr. Ree now is research professor of chemistry at the University of Utah and an expert on reaction processes.

Dr. Eyring, who is dean of the University of Utah graduate school, said Sang recently completed a paper "of fundamental importance" on an old question in science: What makes a jelly set? His research, the dean said, has implications for not only the petroleum industry but for the plastic and textile industries as well.

Many plastics, for example, have, in liquid form, the properties of a jelly. Manipulation of these properties can have an important effect upon the usefulness of the plastics

Hahn

in making injection moldings—the production-line method commonly used in shaping plastic products.

The same properties in a lubricating grease, Dr. Eyring said, effect the ease with which the lubricant can be squirted into tight bearings.

In expressing gratitude to the National Lubricating Grease Institute for establishing its fellowship at the University of Utah, Dr. Eyring said the grant represents a tribute to the expanding research program on the Salt Lake campus. He said Sang Joon Hahn has proved a worthy selection for the grant. "He is both highly intelligent and industrious," the dean said.

The English language, often a barrier to foreign students studying in the United States, has been no problem for Sang. He served United Nations forces as an interpreter and translator during the Korean War.

Graduate of Seoul University

He was a 1948 graduate in chemistry from the University of Seoul, where he studied English. He was a teaching assistant in the College of Agriculture on the Seoul campus when the Korean fighting started. In the early months of the war he was an interpreter for the U. S. Eighth Army's famous Second Division. He later joined the South Korean Marine Corps, which put him to work translating American military science manuals from English into Korean.

In his boyhood he attended public schools in Seoul. Later he studied in the Japanese national high school at Kyoto, where he met Dr. Ree. Dr. Ree then was a professor of chemistry at the University of Kyoto. Dr. Ree later studied and worked with Dr. Eyring at Princeton University.

After the war, Sang taught chemistry at the Ewha University for women at Seoul. It was this position he left in 1955 to begin graduate work at the University of Utah.

KEL-F No. 90 Grease

A Chemically and Thermally Stable Florocarbon Lubricant

THE LUBRICATION OF valves, plug cocks, pumps, agitators and other process equipment under extreme thermal and corrosive conditions has long been a problem to engineers. The inherent stability of fluorocarbons, and in particular chlorofluorocarbons, in contrast to conventional hydrocarbon lubricants under these conditions, makes them a natural choice as lubricants in critical applications. A new type of grease based on "KEL-F" oils having the general formula $Cl\text{--}(CF_2CFCl)_x\text{—}Cl$ has been developed. The following paper describes the physical and chemical properties of "KEL-F" No. 90 grease and gives a preliminary account of its performance in the chemical industry.

Physical Properties

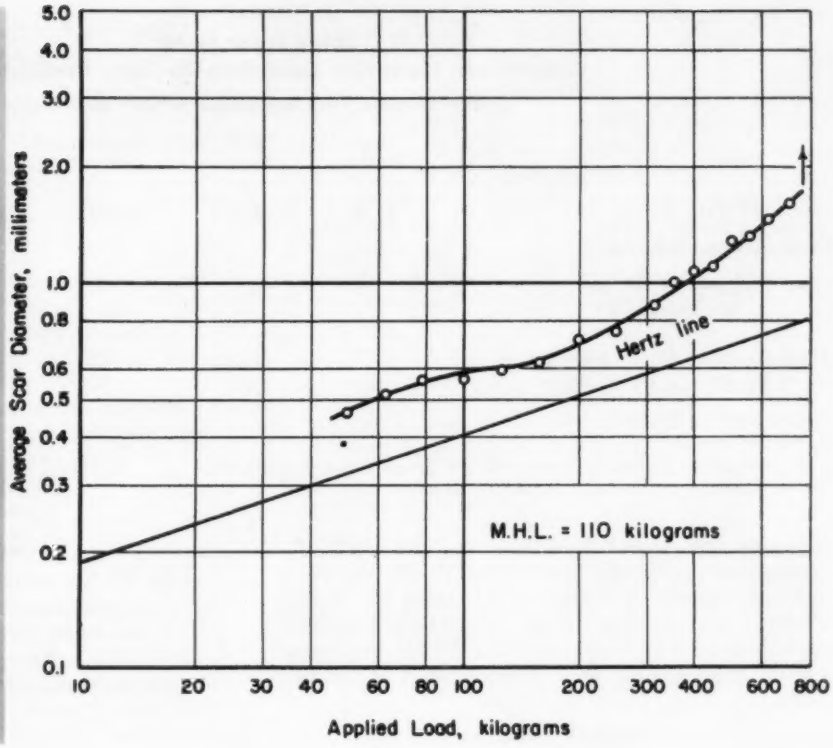
"KEL-F" No. 90 grease has been formulated as a multipurpose lubricant with an NLGI consistency number of 3, equivalent to an ASTM worked penetration of 220-250 mm/10.

Mechanical Stability

The mechanical stability of "KEL-F" No. 90 grease under shear is excellent as shown by tests in the Shell roll apparatus and the Hain micro worker. In the Shell test, a 100 gram sample was kneaded with an 11 pound roller in a steel can turning at 160 rpm. After four hours there was no change in the original ASTM penetration of 240 mm/10; after six hours the penetration had increased but little to 260 mm/10. In the Hain mi-

By R. J. SEFFL, F. W. WEST and F. J. HONN, Kellogg Company

FIGURE 1 illustrates full wear-load diagram in one minute runs when the E. P. properties of KEL-F No. 90 were determined in a Shell tester.



cro worker[1] a 10 gram sample was passed 100 times through a 250 mesh screen, a shearing action equivalent to 100,000 strokes in the standard ASTM grease worker. After this treatment, the consistency decreased only four points from 62 to 58 in the Kaufman micropenetrometer.[2] This change was within experimental error.

Dropping Point

"KEL-F" No. 90 Grease is thickened with an inorganic non-soap gelling agent. As a result, the grease is essentially non-melting with a dropping point of approximately 510°F.

Oil Separation and Evaporation

The amount of oil which separates from "KEL-F" No. 90 Grease at elevated temperatures is small. Tests conducted in accordance with Federal Specification VV-L-791-321.1 show an oil separation of only 2.2% after 120 hours at 212°F., and 7.9% at 300°F. Oil separation after long standing at room temperature is nil.

The fluorocarbon oils and waxes in "KEL-F" No. 90 Grease are sufficiently high in molecular weight that the vapor pressure of the grease at 77°F., is less than 10^{-3} mm. Hg. and oil evaporation amounts to only 2.2% in 125 hours at 212°F. At the same time, the oil base is fluid enough to permit operation as low as 0°F. The over-all recommended temperature range is 0° to 350°F., with intermittent, hot spot operation possible up to 450°F. In the latter case, the life of the lubricant is limited by the volatility of the oil and not by its thermal sensitivity.

Lubricating Properties

"KEL-F" No. 90 Grease is a lubricant with very good extreme pressure (E.P.) properties. The chlorine atoms in the basic $Cl-(CF_2-CFCl)_x-Cl$ structure are believed to be responsible for its lubricity, just as the fluorine atoms are responsible for its heat and chemical resistance.

The E.P. properties of "KEL-F" No. 90 Grease were determined in a Shell Four Ball E.P. Tester. In 10 second runs, the Mean Hertz Load was 113 kg. In one minute runs, the full-wear-load diagram was established (Figure 1). In the latter case, no welding occurred up to the capacity of the machine ($>$ 700 kg.).

Additional lubricity tests were carried out in the Falex and Shell Four Ball Wear testers (Tables 1 and 2). In the Falex machine, excellent results were observed for "KEL-F" No. 90 Grease itself (weld $>$ 2500 lbs.). The performance of a commercial, graphite filled silicone grease was also markedly improved by the incorporation of "KEL-F" No. 90 Grease. The two greases mixed easily in all proportions to give smooth, homogeneous products with lu-

TABLE 1

Falex Wear Tester

One-Minute Transition Load Step-Up Test, Duplicate Runs

(210°F: Standard Steel test pieces: 94°-95° Block angle)

Grease	*Wear, No. of Teeth*[1] *Min.*	*Max.*	*Seizure Load*[2] *lbs. gauge*	*Weld Load lbs. gauge*	*Avg. Torque lbs.-in.*
"KEL-F" No. 90	0	8	NS	>2500	16
Dow Corning Silicone 41[3]	–	–	–	< 300	–
Dow Corning 41, 85 wt. %	6	6	NS	500	23
+ "KEL-F" No. 90, 15 wt. %	–	–	–	< 500	19
Dow Corning 41, 15 wt. %	2	11	NS	>2500	19
+ "KEL-F" No. 90, 85 wt. %	3	12	NS	>2500	19

1. One tooth=0.0000556 inch of wear.
2. NS=no seizure.
3. A commercial high-temperature silicone grease containing graphite.

bricating properties dependent on the "KEL-F" Grease content. In the Shell Wear Tester, the average scar diameter was noticeably smaller for "KEL-F" No. 90 Grease than for the graphite-filled silicone. As little as 15% by weight of "KEL-F" No. 90 Grease greatly improved the wear characteristics of the silicone.

Chemical Properties

As a fluorocarbon, "KEL-F" No. 90 Grease is resistant to the attack of a wide range of acids, alkalies and oxidants. Yet, the grease can be completely removed for cleaning purposes by common organic solvents.

Water Resistance

"KEL-F" No. 90 Grease is resistant to water washout and breakdown. When tested in accordance with Federal Specification VV-L-791-3252.1, the water washout was only 9%, which is much better than the value observed for soap-gelled hydrocarbon lubricants.

No cloudiness was noted in the 10 minute MIL-G-3278 water stability test.

TABLE 2

Shell-Four Ball Wear Tester

600 rpms. 7 kgload: 2 Hours: Steel Balls[1]

Grease	*Temp. °F.*	*Avg. Scar Diam., mm*	*Avg. Torque, g-cm*
"KEL-F" No. 90 Grease	176	0.498	476
Dow Corning Silicone 41	176	0.842	[2]
Dow Corning 41, 85 wt. %	176	0.512	249
+"KEL-F" No. 90, 15 wt.%	356	0.590	264

1. Precision Scientific Company, Part No. 75017, Lot 30L31, as used in the Shell Four-ball EP tester.
2. Test terminated after 17 minutes because of excessive squealing and vibration.

Metal Corrosion

"KEL-F" No. 90 Grease is non-acidic and non-corrosive toward metals. It does not stain a copper corrosion strip when tested for one hour at 212°F. (VV-L-791-5309.1). One precaution should be observed. The grease should not be used to lubricate aluminum-on-aluminum where large shear forces are involved. Under shear, the thin oxide layer normally present on the surface of aluminum is apparently wiped away and catalytic amounts of aluminum halide are formed which in conjunction with high interface temperatures rapidly decompose the $Cl(CF_2–CFCl)_xCl$ structure. In the absence of shear, however, aluminum can be heated to 400°F., without interacting with "KEL-F" Grease.

Oxygen Stability

An outstanding feature of fluorocarbons is their resistance to oxidation by oxygen, ozone, hydrogen peroxide, chromic acid, nitric acid, and other powerful reagents. The fluorocarbons are also resistant to the natural forces of atmospheric deterioration, which are usually oxidative in character. The resistance is an inherent property of the C-F structure, since fluorine is the most electronegative element in the periodic table.

In the Norma-Hoffman oxygen bomb test (ASTM Designation D942-50), at 212°F. and 110 psig. oxygen pressure, there was no pressure drop after 268 hours. Under more severe conditions, two hours at 2000 psig. and 300°F., there was no apparent change in the grease.

Performance in Chemical Industry

Recently, samples of "KEL-F" No. 90 Grease were distributed to nearly 600 laboratories in the chemical and allied industries for evaluation in some of the most troublesome lubrication applications. A partial

TABLE 3
Successful Chemicals Reported for "Kel-F" No. 90 Grease

Oxidizing Agents	*Halogens*
Fuming Nitric Acid	Fluorine
Hydrogen peroxide	Chlorine
Oxygen	Bromine
Chromyl chloride	Iodine monochloride
Chromyl nitrate	Bromine trifluoride
Potassium permanganate	
Strong Acids and Corrosive Chemicals	*Miscellaneous Chemicals and Solvents*
Conc. sulfuric acid	Aqueous acids and bases
Hydrogen fluoride	Alcohol
Hydrogen chloride	Ether
Sulfan (sulfur trioxide)	Hydrocarbon solvents
Chlorosulfonic acid	Chlorinated hydrocarbons
Antimony trichloride	Fatty acids
Phosphorus oxychloride	Silanes
Aluminum chloride	Furanes
Trifluoromethyl hypofluorite	
Nitrogen oxyfluoride	
Nitroxyl fluoride	

list of chemicals with which successful performance was reported is shown in Table 3.

Applications

In the laboratory "KEL-F" No. 90 Grease* is being used to lubricate glass joints and stopcocks in contact with acids, alkalies, oxidants, and halogens. In the plant, the grease is finding application in plug cocks, valve stems, threads, packing glands, bearings, O-ring seals, agitator and pump shafts and many related types of equipment where heat and chemical resistance must be combined with lubricating properties.

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2. Kaufman, G., Finn, W. J., and Hanington, R. F., *Ind. Eng. Chem., Anal. Ed.,* 2 108-10 (1939).

*Registered trademark of the M. W. Kellogg company for its fluorocarbon products.

Effective April 1, 1957, all KEL-F fluorocarbon products including No. 90 grease, will be manufactured and sold by the Jersey City Chemical Division, Minnesota Mining and Manufacturing company, Jersey City 3, New Jersey.

About the Authors

FRANCIS J. HONN *is a graduate of John Carroll University with a B. S. degree, and received his M. S. degree from Brooklyn Polytechnic Institute and a Ph. D. from the University of Pittsburgh. Honn has served as a research chemist with the General Cable corporation, a fellow at the Mellon Institute, and a research chemist with the M. W. Kellogg company where he is now head of the applications laboratory. He is a member of New York Chemists Club, ACS and ASTM.*

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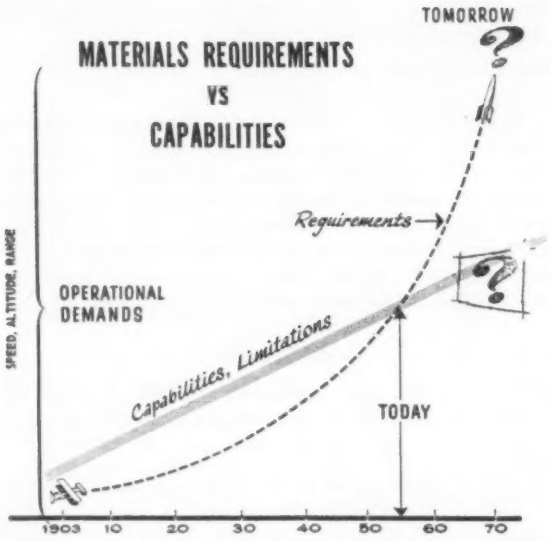

FIGURE 1. Where we have been and where we are going in terms of materials — requirements exceed capability.

Materials the Key to Aeronautical Progress

By E. M. GLASS
Wright Air Development Center

I WOULD LIKE to give you a few tantalizing tips, and possibly a couple of jolts at the same time. All of you undoubtedly have heard the slogan, "As Maine goes, so goes the nation." I realize that this is quite a controversial and debatable statement. But I doubt whether any of you disagree with the following statement: "Materials are the Key to Aeronautical Progress." As materials go, so go our air weapons. Materials are actually the backbone of all engineering structure and machines. Every major improvement in the performance of airplanes and engines resulted from the development of new materials or from the reprocessing of old materials into new products.

In discussing this question of materials, I would like to point out that we in the Air Force consider lubricants to be just as important a structural material as we do a metal or plastic, and while I may not cover specifically many of the aspects of lubrication in this talk, it is just as applicable to lubrication and lubricants.

Figure 1 is an attempt to provide a very broad picture of where we have been and where we are going in terms of materials in general. During and since World War II, materials research and development has proceeded in an orderly and modest fashion, and significant, but gradual, progress has been achieved. But during this same time, with the rapid exploitation of the gas turbine engine, ram jets, the rocket engines, supersonic flight, and complex electronic devices, materials requirements have actually increased exponentially. This has led to the disheartening situation we are faced with today where materials requirements actually exceed capability.

Major factors responsible for this situation are the more intensified and new environments within which air weapons must exist. The thermal problem is one

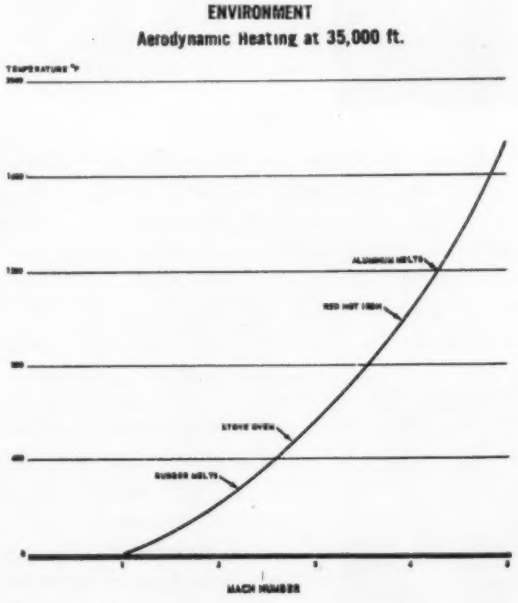

FIGURE 2. Illustrates the thermal problem. Note that melting point of aluminum is between mach 4 and mach 5.

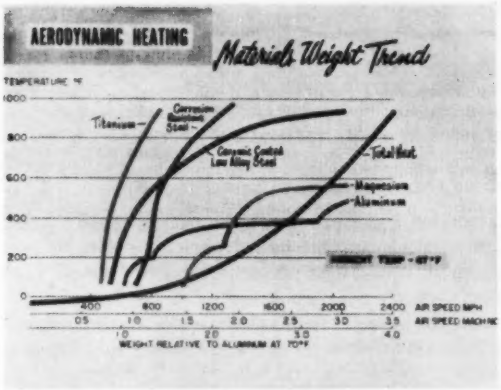

FIGURE 3. Emphasizes importance of strength/weight ratio of structurals. Aerodynamic heating curve in Figure 2 is superimposed. Trend is away from aluminum.

with which you are all familiar. I noted with great interest the thermal theme throughout the NLGI meeting and the CRC symposium. In addition to the geographical, chemical and high altitude environments, we must now learn to live with a relatively new and perplexing environment – radiation. Enthreaded through all of these problems is the element of time– the timely development of materials to meet the performance requirements of future air weapon systems.

During this discussion I would like to concentrate on the heat problem, the radiation problem, and dwell for a bit on the problem of time.

Let's look at the thermal problem first, illustrated in Figure 2. This chart was prepared to provide a few reference points based on commonly known materials. Here we can appreciate the temperature build-up resulting from aerodynamic heating at an altitude of 35,000 feet. Note that between mach 4 and mach 5, the melting point of aluminum is attained. We have reached that point in time where aluminum, which has been the basic aeronautical material of construction since the middle 1920's, is now becoming obsolete.

Figure 3 amplifies this further and emphasizes the important consideration of strength/weight ratio of structural materials. This figure plots strength/weight ratios of some of our more common structural materials as a function of temperature. Super-imposed upon this is the aerodynamic heating curve in Figure 2. It becomes immediately apparent why the trend in structural materials is away from alumnium and to titanium and stainless steel. For the specific design upon which these curves were based, an aluminum structural member would require three to four times the weight required for a comparable titanium or steel structure at 400°F.

It appears appropriate at this time to cover briefly some of the lubrication problems of the future. Figure 4 summarizes the trends of speed and temperatures of accessory equipment such as alternators and auxiliary

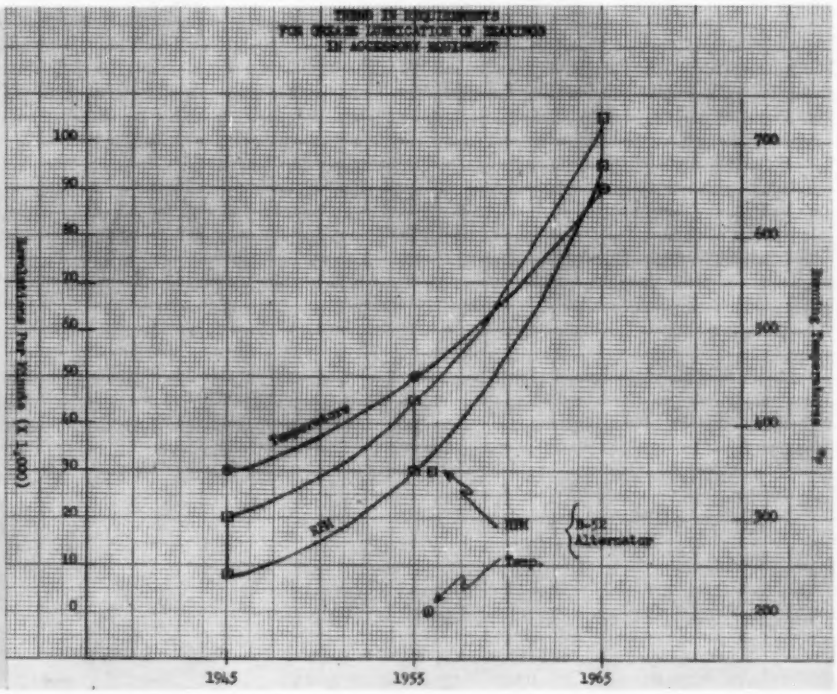

FIGURE 4. Summary of trends of speed and temperatures of accessory equipment — alternators and auxiliary power units — of ascending importance in future weapons field.

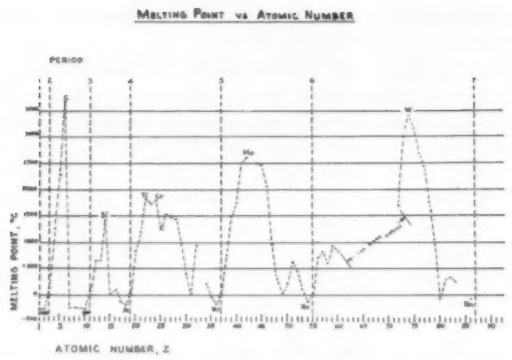

FIGURE 5. Plotting of melting point against atomic number represents the true "materials barrier" facing Air Force. Material ability alone is no longer effective.

power units. This class of equipment was selected because of its ascending importance in future weapon systems and the interest of both the NLGI and the CRC. These curves represent the composite prognostications of accessory designers, bearing engineers and people associated with the development of lubricants. They are based primarily on the anticipated state of the art for the next five to ten years. I sincerely hope that you prove us pessimistic.

Until now we have been concerned primarily with aerodynamic heating problems posed by the more conventional air weapons. Some of the less conventional air weapons pose aerodynamic heating problems which make these problems look relatively simple. For example, under specific hypersonic conditions, it is not inconceivable that airfoils could be required to withstand a heat imput of as much as 10,000 BTU's/sq. ft./sec. Translating this into temperatures, this would be in the neighborhood of 5000-8000°F. In addition to the aerodynamic heating problem, there is the problem of heat in and from propulsion devices. Calculations based on ram jets and rockets operating under hypersonic conditions indicate that engine temperatures exceeding 7000° F may be experienced. On this basis, the thermal environment we are now using as a basis for development and tests are relatively mild, compared to what the future, say the next ten to twenty years, actually holds. Fortunately, in many cases, time at temperature is extremely short.

After viewing these fantastic temperatures requirements of the future, it might be appropriate to take a look at what we have in the materials "bank." Figure 5 plots melting point against atomic number. This represents the true "materials barrier" facing the Air Force. As we push closer and closer to ultimate in materials capability, it becomes increasingly clear that we can no longer depend solely upon the "brute force" ability of a material to solve these problems. We must bring together the best possible materials and the necessary knowledge and designs to take advantage of their unique properties while minimizing their deficiencies.

Why must we have to worry about these temperatures? Why can't the designers find some means of cooling airfoils and equipment. The Air Force is naturally attacking this problem also. But to cool generally means added weight. Figure 6 gives us a measure of the magnitude of the weight problem. As serious as the problem is in aircraft, it is intensified greatly in guided missiles. We are caught in a squeeze between the "materials barrier" on the one hand and the weight limitation on the other. This fact emphasizes further that the designer must learn to take advantage of the unique properties of available materials. Those associated with materials development must provide materials with the best possible properties and the design criteria which will foster more efficient design use of these materials.

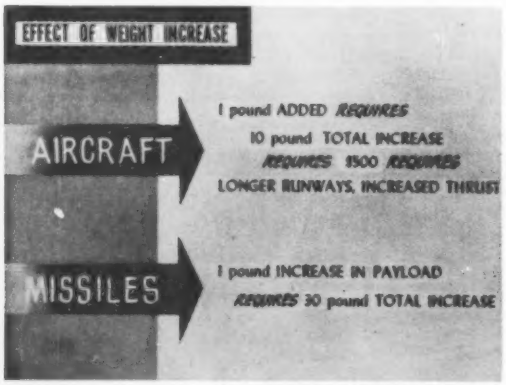

FIGURE 6. A measure of the magnitude of the weight problem in aircraft and its magnification in missiles.

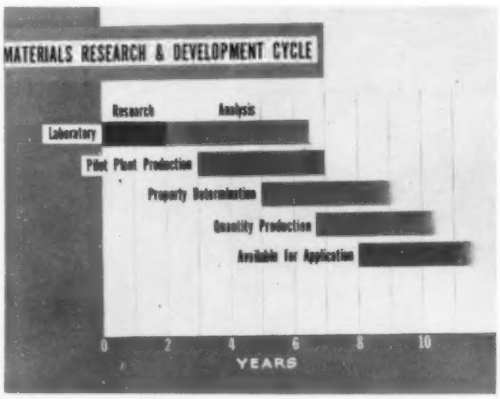

FIGURE 7. Illustrates time it takes to go from the laboratory to application of a material in production

Until now we have concerned ourselves primarily with the thermal environment. All of you are no doubt aware of the Air Force's interest in nuclear propulsion as embodied in weapon system 125A. While the biological effects of radiation have been publicized widely, little mention has been made on the effects of radiation on materials.

As reflected in the exhibits at the symposium, the effects of radiation on some typical materials were displayed. Butyl rubber turned into a gummy mass completely devoid of usable properties. Laminated plastics of the type used in radomes discolored slightly but essentially retained their properties. Laminated transparent plastics used in aircraft canopies formed bubbles which would seriously affect visibility. Greases such as the substituted aryl ureasilicone grease exhibited excellent rubber-like properties. Hydraulic fluids and engine oils solidify.

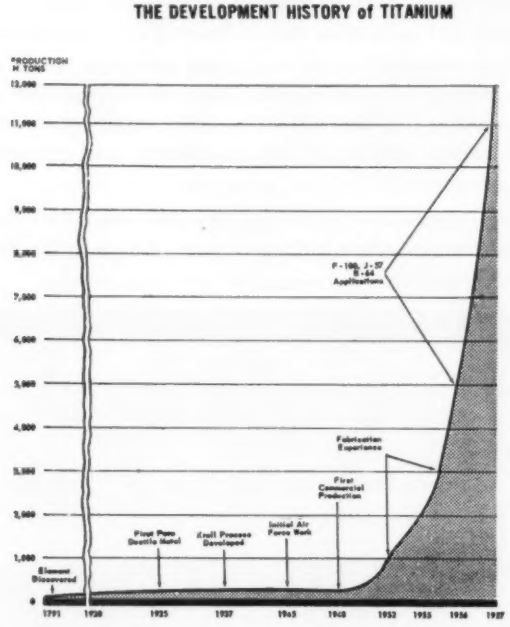

FIGURE 8. Development cycle of a material is not completed with the laboratory demonstration of feasibility.

Future weapon systems with nuclear power plants will require materials which are both thermal and radiation resistant.

So far we have discussed the nuclear and thermal problems. Some mention should be made of the problem of lead time. *Figure* 7 illustrates the time it takes to go from the laboratory to application of a material in production. Before a material can be selected for an application there must be adequate assurance that this material will be available for use in adequate quantities and the quantity production will reflect uniformly the required properties. Thus the development cycle of a material is not completed with the laboratory demonstration of feasibility. This point is illustrated further in *Figure* 8. As discussed previously, optimum design must be based on the unique properties of a material. Mere substitution of one material for another generally provides marginal improvements only. If we are to provide the materials required for the weapon systems of the 1960-65 period, we must learn how to reduce the long lead time now required from development to application. Until this is possible, materials lead time must be considered a major factor in scheduling future air vehicles.

In summary, an attempt has been made to give you what the military briefers call "The Big Picture." We have reviewed in general some of the challenges facing all of us; the lead time of five to eight years from the time a material is developed to the time it can be applied to a particular weapon system. This is not solely the Air Force's problem, it is industry's problem as well. The Air Force has depended almost entirely upon industry to help solve these particular problems. The CRC has been doing yeoman's work in providing the Air Force with tools on which future developments can be based and which give us a better understanding of the behavior of lubricants. It is hoped that this presentation will have provided some impetus to your work, and assist you in focusing your sights on the new horizons of the Air Force. ■

Presented at the 1956 *Coordinating Research Council*

About the Author

EDWARD M. GLASS *is chief of the operations office of the Wright Field Materials Laboratory, Wright Air Development Center. This position encompasses both planning future programs and the direction of current research and development programs. A graduate of Rhode Island State, with a Master's degree from Virginia Polytechnic Institute, Glass entered the War Department in* 1942 *and was assigned to Wright Field as an aircraft maintenance supervisor, Air Service Command. He was appointed assistant chief in the chemical branch of Wright's petroleum products section and assigned to the Materials Laboratory staff in* 1950.

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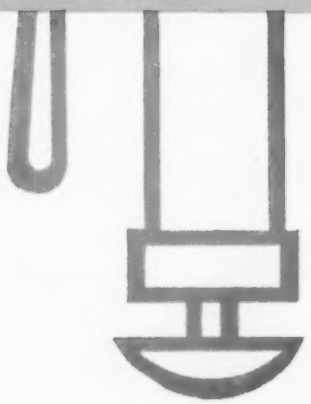

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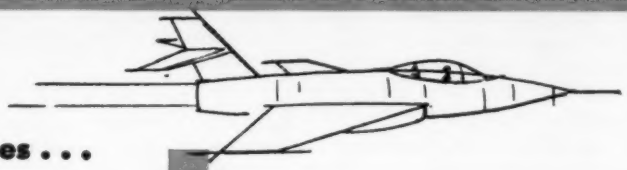

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Patents and Developments

Amate-Dicarboxylate-Thickened Grease

U. S. Patent No. 2,756,213 to California Research corporation. Grease compositions thickened with amic acid salt-dibasic acid salt mixtures and claimed to remain unctuous and not become hard or brittle in the range of 500°F. The mixtures are prepared by reacting a mol of a primary amine (e.g., lauryl amine) with at least 1.5 mols of a dibasic acid (e.g., sebacic acid), which mixture then is reacted with NaOH or similar base, producing the amic acid salt-dibasic acid salt mixture (e.g., N-lauryl-sodium sebacamate). Small amounts (e.g., less than 6%) can be used to thicken lubricating oils.

DETAILS

It is becoming more and more apparent that, for the most part, greases must be able to lubricate effectively under conditions of temperatures in excess of 400°F., and, in certain instances, in the range of 500°F. Numerous military and industrial grease specifications describe greases having dropping points of 400°F. as a minimum. This need for high temperature greases is the result of increased speeds of gears, bearings, and other moving parts; increased pressures caused by the development of smaller gears to withstand greater loads than heretofore expected of larger gears, etc.

In the automotive industry, for example, there is the constant desire to manufacture smaller internal combustion engines without sacrificing power output. In fact, many of these smaller engines are designed to deliver greater power than their larger counterparts. The same smaller engines have considerably less bearing surfaces than the larger predecessors, which means that the bearings gear greater loads than before.

Similarly, the continuing trend to manufacture automobiles with lower centers of gravity has made it necessary to use smaller gears, particularly in such gear assemblies as the differentials and transmissions. The smaller gears thus used have considerably greater pressures exerted upon them per unit than the older type gear assemblies. Thus, machines having higher loads on bearings and gears along with greater speeds, require grease compositions which will function at higher temperatures than heretofore necessary.

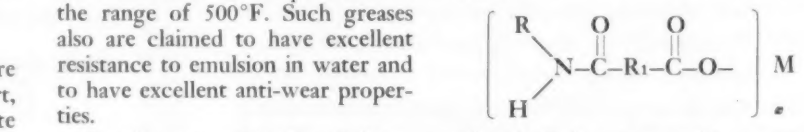
U. S. Patent 2,756,213, issued to California Research corporation, discloses grease compositions thickened with amic acid salt-dibasic salt mixtures, which are claimed to remain unctuous and not become hard or brittle at temperatures in the range of 500°F. Such greases also are claimed to have excellent resistance to emulsion in water and to have excellent anti-wear properties.

Certain amic acid salts of themselves thicken lubricating oils to the consistency of greases. Such amic acid salt thickening agents have been prepared from amic acids obtained by reacting stoichiometric amounts of amines and dibasic acids; that is, 1 mole of a primary amine (e.g., lauryl amine) was reacted with 1 mole of dibasic acid (e.g., sebacic acid) to form the desired amic acid (e.g., N-monolaurylsebacamic acid). This amic acid was then reacted with a metal hydroxide (e.g., sodium hydroxide) to form the sodium soap of the amic acid (e.g., N-laurylsodium sebacamate) which was used as a grease thickening agent. Such amic acid salts of themselves are used in the preparation of greases having dropping points in the region of 400°F. However, they are ineffective in the preparation of greases having dropping points in the region of 500°F.

Dibasic acid salts (e.g., sodium sebacate) of themselves exhibit no gelatin effect when heated in lubricating oils. That is, when dibasic acid soaps are mixed with lubricating oils, and such mixtures are heated to high temperatures (e.g., 500°F.), cooled, and milled, no gel structure nor grease is obtained.

The amic acid salt-dibasic acid salt mixtures of this patent are prepared by reacting 1 mol of a primary (or secondary) amine with at least 1.5 mols of a dibasic acid to form an amic acid-dibasic acid mixture; which mixture is then reacted with a metal oxide or hydroxide, resulting in an amic acid salt-dibasic acid salt mixture.

The amic acid salts which are present in the grease thickening composition described herein are represented by the following formula

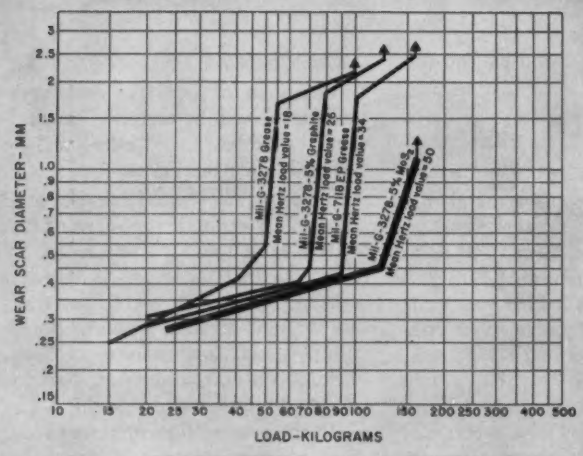


wherein R is a saturated or unsaturated, straight-chained, branch-chained or cyclic essentially hydrocarbonaceous radical having from 2 to 50 carbon atoms (8 to 20 preferred), R_1 is a saturated or unsaturated straight-chained, branch-chained, or cyclic essentially hydrocarbonaceous radical having from 1 to 54 carbon atoms, M is a metal, and x is a number having a value equal to the valence of the metal M.

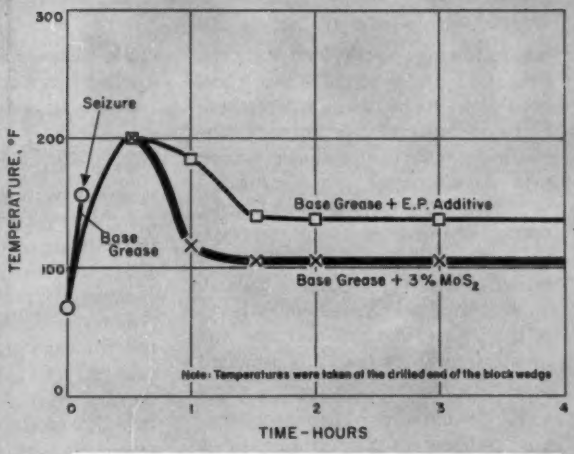
It is preferred that R be a straight-chained or cyclic essentially hydrocarbonaceous radical and that R_1 be a straight-chained essentially hydrocarbonaceous radical.

Examples of the R radicals, include the following: ethyl, propyl, propenyl, butyl, butenyl, pentyl, hexyl, hexenyl, octyl, octenyl, nonyl, decyl, decenyl, dodecyl, dodecenyl, tetradecyl, tetradecenyl, hexadecyl, octadecyl, octadecenyl, eicosyl, triacontyl radicals, radicals derived from petroleum hydrocarbons, such as white oil, wax, olefin polymers, e. g., polypropylene and polybutene, etc.

Examples of R_1 radicals include


WEAR SCAR DIAMETER - MM
LOAD - KILOGRAMS
Mil-G-3278 Grease
Mean Hertz load value = 18
Mil-G-3278 - 5% Graphite
Mean Hertz load value = 26
Mil-G-7118 E.P. Grease
Mean Hertz load value = 34
Mil-G-3278 - 5% MoS2
Mean Hertz load value = 50

4-ball E.P. test shows the superior extreme pressure properties of MoS_2 grease over the same grease without additives. In the same test, grease containing 5% graphite had a mean Hertz load value of 26; the conventional E.P. grease Hertz value was 34.


TEMPERATURE, °F
TIME - HOURS
Seizure
Base Grease + E.P. Additive
Base Grease
Base Grease + 3% MoS2
Note: Temperatures were taken at the drilled end of the block wedge

Timken endurance test clearly shows how MoS_2 functions when added to a base grease. With base grease alone, the specimen seized in about 10 minutes. With MoS_2 added, lubrication was effectively sustained throughout the test at a reduced temperature of about 100°, compared with a temperature of about 130° for the conventional E.P. grease.

Moly-Sulfide additive extends effective lubrication

Laboratory and field tests prove that chassis lubricants containing MoS_2 sustain lubrication after base grease is wiped off

When standard chassis greases are first applied to shackle pins, ball points and other bearing points under trucks, trailers and passenger cars, excellent results are obtained. These greases, however, are subject to mechanical motion, such as reciprocation, pressure, oscillation and shock loading which tend to eject the lubricant.

At this point, chassis grease containing 3% Moly-Sulfide proves its great value. MoS_2 is forced between the rubbing surfaces, adheres to the metal and forms a protective film that prevents galling, welding and fretting. The extent to which MoS_2 sustains lubrication is demonstrated by substantial reduction in wear.

Four tests show how MoS_2 reduces wear

1. **Shell 4-ball extreme pressure test** (see illustration) —MoS_2 addition reduces wear more than 50%, increases load capacity about 70% over base grease.

2. **Falex 2-hour wear test**—As the chart here shows, MoS_2 sustains lubrication, as proved by a reduction in wear, by an average factor of 500.

Grease	Loss in weight of specimen
Lithium-base	1,000 mg.
Lithium-base plus 2% MoS_2	1.98 mg.—average of 3 runs

3. **Timken endurance test** (see illustration)—MoS_2 sustains lubrication well beyond the capabilities of the base grease alone, and at a lower temperature than conventional E.P. grease.

4. **Simulated shackle bolt service test**—oscillating friction machine—3% MoS_2 addition virtually doubles the effective lubrication of a lithium base chassis grease —is far more effective than conventional additions.

Hours to failure—average of 2 tests

Additive	None	3% MoS_2	5% ZnO	15% ZnO
Dry	350	564	336	555
Wet	162	264	109	85

On-the-road use proves MoS_2 prevents chassis wear

A St. Louis company reports no noticeable wear on chassis parts of auto transport trailers, since they selected an MoS_2 chassis grease.

Based on their own tests, several major automobile and truck manufacturers use—and recommend—MoS_2 grease for torsion suspension units, ball joint suspension, as well as for many other critical-wear areas.

Moly-Sulfide greases are available from many producers today. For a list of the manufacturers of these greases—which includes several major oil companies—and for a copy of "Molybdenum Disulfide as a Grease Additive," write to Department 58, *Climax Molybdenum Company, 500 Fifth Avenue, New York 36, N. Y.*

the following: methylene radicals, i. e., (CH_2) x, wherein x is a number from 1 to 52 (6 to 24 preferred; 12 to 18 especially preferred) (including methylene radicals which may have one or more saturated or unsaturated, straight-chained, branch-chained, or cyclic essentially hydrocarbonaceous radical attached thereto); a phenyl radical, a substituted phenyl radical (wherein the phenyl nucleus contains one or more aliphatic radicals attached thereto); etc.

By "essentially hydrocarbonaceous" radicals is meant those radicals which are composed mainly of hydrogen and carbon and include such radicals which include, in addition, minor amounts of the substituents such as chlorine, bromine, oxygen, nitrogen, etc.

The dibasic acid salts present in the grease thickening mixtures include those represented by the following formula:

$$\left[-O-\overset{O}{\overset{\|}{C}}-R_2-\overset{O}{\overset{\|}{C}}-O- \right]_x M$$

wherein M represents a metal, x is a number having a value equal to the valence of the metal, and R_2 is a saturated or unsaturated, straight-chained, branch-chained or cylic essentially hydrocarbonaceous radical; for example, a methylene radical, i.e., (CH_2) x, wherein x is a number from 1 to 52 (4 to 16 preferred; 4 to 10 especially preferred) (including such methylene groups which may have one or more saturated or unsaturated, straight-chained, branch-chained or cyclic essentially hydrocarbonaceous radicals attached thereto); a phenyl group, a substituted phenyl radical (wherein the phenyl nucleus has one or more aliphatic radicals attached thereto); etc.

For sake of brevity, the "amic acid salts" will be termed "amates," and the "dibasic acid salts" will be termed "dicarboxylates."

The metals which can be used in the formation of the soaps of this invention include the metals of groups I, II, III, and IV of Mendeleff's periodic table. Particular metals include lithium, sodium, potassium, silver magnesium, calcium, zinc, strontium, cadmium, barium, aluminum and lead.

In the preparation of such grease thickening agents, dicarboxylic acids were used which had been prepared by polymerizing unsaturated monobasic acids. These dicarboxylic acid preparations are known as dimer acids, and they are obtained from Emery Industries, Inc., Cincinnati, Ohio. These dicarboxylic acid preparations con-

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Saponifiable	94%
Free Fatty Acid (as oleic)	55-60%
Actual Free Fatty Acid Content	90%
Saponification No.	120-130
Free Inorganic Acid	0.2% max.
Iodine Value	20-40
Apparent Solidification Point (titre)	Approx. 44° C.
Softening Point	45-48° C.
% Sulfur	No corrosive sulfur

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tain a small amount of tricarboxylic acids (approximately 12%), which also form grease-thickening agents when reacted with primary amines, followed by reaction with a metal oxide or hydroxide to form the amate-dicarboxylate-tricarboxylate grease-thickening agent.

Example.—Preparation of grease thickened with lithium-N-hexadecyl dilinoleamide-lithium dilinoleate

300 grams (1.5 moles) of dilinoleic acid[1] were heated to 300°F., after which 136 grams (0.5 mole) or armeen HTD[2] were added dropwise during a period of 15 minutes. The reaction mixture was heated, with stirring, at a temperature range of 400°F. to 420°F. until all of the water of reaction had been removed. Upon cooling, the reaction product was a dark sticky solid having a neutralization number of 64.

A mixture of 15 grams of the above solid reaction product and 85 grams of a California solvent-refined naphthenic base oil having a viscosity of 450 SSU at 100° F. was charged to an 800 ml. Pyrex beaker. This mixture was heated, with stirring, to 200° F., then an aqueous solution of 1.44 grams of $LiOH\text{-}H_2O$ was added. The mixture was heated to a temperature of 390 F. After cooling, the product was a clear amber grease.

In the above examples of the grease preparations, the salts were prepared "in situ"; however, the salts may also be prepared as separate products, then dispersed in oils to form greases. The amic acids themselves may also be prepared directly in the oil.

Table I hereinbelow presents data on the use of amate-dicarboxylate mixtures as grease thickeners according to this patent. The thickening agents were prepared by reacting various primary amines with adipic acid, followed by reaction with a metal hydroxide. With the exception of greases 9 and 10, which respectively contained 24% and 14%, all of the greases contained 16%, by weight, of the thickening agent.

The various symbols used in Table I are explained as follows:

"HTD" refers to Armeed HTD, a product of Armour and Company, Chicago, Illinois, containing 25% n-hexadecylamine, 70% n-Octadecylamine, and 5% n-octadecenylamine.

"T" refers to armeen T, a product of Armour and Company, Chicago, Illinois, containing a mixture comprising 30% hexadecylamine, 25% octadecylamine and 45% hexadecylamine. 85% of this mixture consists of n-primary amines.

"C" amine refers to armeen "C," which contains 8% octylamine, 9% decylamine, 47% dodecylamine, 18% tetradecylamine, 8% hexadecylamine, 5% octadecylamine, and 5% octadecenylamine.

"TD" refers to armeed TD,

which is a purified product of the above armeed "T." The "TD" product contains 30% n-primary hexadecylamine, 25% n-primary octadecylamine and 45% n-primary octadecenylamine.

"HT" refers to armeed HT, which is a mixture of amines comprising 25% hexadecylamine, 70% octadecylamine and 5% octadecenylamine. 85% of this mixture consists of n-primary amines.

Base oil "A" was a California solvent-refined naphthenic base oil having a viscosity of 450 SSU at 100° F.

Base oil "B" was a California solvent-refined paraffinic base oil having a viscosity of 480 SSU at 100° F.

Base oil "C" was a di(2-ethylhexl) sebacate.

Base oil "D" was a poly(methylphenyl) siloxane.

Base oil "E" was a California solvent-refined naphthenic base having a viscosity of 90 SSU at 210° F.

[1]This dilinoleic acid is essentially a dibasic acid of approximately 600 molecular weight of Emery Industries, Inc., Cincinnati, Ohio. It is prepared by the dimerization of linoleic acid. Although the product is essentially a dibasic acid, approximately 12% of trilinoleic acid (a tribasic acid) and 3% of linoleic acid (a monobasic acid) are present.

[2]Armeed HTD is a product of the Armour company, Chicago, Illinois, and contains 25% of n-hexadecylamine 70% of n-octyldecylamine, and 5% of n-octadecenylamine. This means molecular weight of this product is 264.

When the amate-dicarboxylate grease thickening agents described herein are prepared from high molecular weight amines and/or high molecular weight dibasic acids, small amounts (e.g., less than 6%) of such amate-dicarboxylate mixtures can be used to thicken lubricating oils. Similarly, when amate-dicarboxylate mixtures are prepared from low molecular weight amines and/or dibasic acids, excellent lubricating oil thickening agents are obtained; that is lubricating oils thickened to the consistency of extremely soft greases or greases having extremely low yield values. Such thickened lubricating oils may be used for filter elements, such as in air filter elements of diesel engines, air-conditioning units in general, etc.

As another embodiment of this patent, a dibasic acid salt may be added to an oil containing an amic acid salt; or a dibasic acid may be mixed with an amic acid before neutralization with a metal. For example, if in the original preparation of the amic acid-dibasic acid mixture, the mole ratio of dibasic acid to amine has a value of 1 or less than 1, additional dibasic acid may be incorporated into the amic acid thus prepared before neutralization with metal hydroxide, or additional dibasic acid salt may be incorporated into the oil prior to the grease preparation. The dibasic acid (or salt) added may or may not be the same as that used in the original preparation.

TABLE I

Experiment No.	*Reactants for Soap Preparation*			*Grease Properties*		
	n-primary amine	*Mole Acid/ Mole Amine*	*Metal Hydroxide*	*Dropping Point, ° F.*	*ASTM Penetrate (Worked 78° F.)*	*Base Oil*
1	hexyl	2	Li	421	360	A
2	cyclohexyl	4	Li	500+	355	A
3	HTD	2	Na	486	195	B
4	HTD	2	Al	431	304	A
5	phenyl	4	Li	...	...	A
6	octadecyl	2	Li	474	350	D
7	T	2	Li	500+	284	A
8	TD	2	Li	500+	360	A
9	HT	2	Li	500+	282	B
10	HT	2	Li	500+	285	E
11	T	2	Li	500+	334	A
12	C	3	Li	500+	313	A

Synthetic Oil Base Greases

U.S. Patent 2,746,924 to Esso Research and Engineering company. Formals having the general formula $(RO)_2CH_2$, where R is a branched chain aliphatic radical having 8-20 carbon atoms, are employed as synthetic oil bases, particularly in conjunction with grease-forming soaps. The formals may be produced by reacting formaldehyde with branched chain alcohols prepared by the oxo synthesis. One example employs 7.5% lithium stearate, 7.5% lithium hydroxystearate, 0.5% phenothiazine and 84.5% formal of C_{10} Oxo alcohol.

DETAILS

In an effort to obtain superior lubricating compositions having specific and unusual characteristics there have been developed new synthetic lubricating compositions. One class of material which has been found to be especially operable as synthetic lubricants is the long chain aliphatic esters, both the simple dibasic acid esters and the complex esters prepared from dibasic acids, glycols, and alcohols. In general these synthetic lubricating oils are characterized by viscosity properties that are outstanding both at low and high temperature when compared to mineral lubricating oils. These outstanding high and low temperature properties are especially desirable for the lubrication of combustion engines which are designed to operate at high and low temperatures. It has been found that mineral lubricating oils are unsatisfactory for lubricating these combustion engines because of their somewhat restricted

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operating temperature range due to the limits of their viscosity characteristics.

U. S. Patent 2,746,924, issued to Esso Research and Engineering company contemplates the formation of a new type of synthetic lubricant which has outstanding viscosity properties and is thereby useful as a substitute for mineral lubricating compositions. These new synthetic lubricants are, so far as is known, new compositions of matter, and comprise the acetals made from branched chain aliphatic alcohols and formaldehyde or a polymer of formaldehyde. Other aldehydes, such as acetaldehyde, may be used. However, formaldehyde is preferred.

The synthetic lubricants of this patent have the following general formula: $(RO)_2CH_2$ where R is a branched chain aliphatic radical having from 8 to 20 carbon atoms.

These formals are very simply prepared in accordance with the following technique.

Formaldehyde in aqueous solution or any of the polymers of formaldehyde, such as paraformaldehyde, is refluxed with the desired branched chain alcohol. Acidic materials such as hydrochloric acid, sulfuric acid, sulfamic acid, p-toluenesulfonic acid, sodium acid sulfate, or any strong acid may be used as a catalyst. Entraining liquids such as hexane, heptane fractions, benzene and the like may be used in order to remove the water of reaction, although good yields are readily obtainable without a water entrainer. The product may be water-washed followed by an alkali wash, and dried over some suitable drying agent, such as anhydrous potassium carbonate, or by distillation using a hydrocarbon as a water entrainer. Distillation may be under reduced pressure if desired.

It is essential that the aliphatic alcohol portion of the formal radical be highly branched in order to satisfy the viscosity requirements for lubricating oil use. Various straight chain formals have been prepared but those of proper chain length have pour points that are too high for ordinary lubrication work. The formal of n-tetradecyl alcohol melts at 107° F. (Chem. Abs. 42, 4922 (1948)), and that of n-nonadecyl alcohol melts at 140° F. (Ber. 72B, 1060 (1939)). The formal of technical C_{16} alcohol, a straight chain, alcohol, was prepared and was a solid at room temperature. A formal of technical lauryl alcohol was a liquid at room temperature but had an ASTM pour point of 65° F., making it entirely unsatisfactory as a lubricating oil at low temperatures.

Particularly desirable branched chain alcohols are those highly branched chain aliphatic alcohols prepared by the "oxo" synthesis. This "oxo" synthesis may be described as being the catalytic reaction product of an olefin with carbon monoxide and hydrogen. The reaction occurs at temperatures in the order of 300°-400° F., at pressures in the range of about 1000 to 3000 p.s.i. In the presence of a suitable catalyst, ordinarily a heavy metal carbonyl such as cobalt carbonyl, there is formed an aldehyde which is subsequently hydrogenated to a primary alcohol. This process was first developed in Germany and is described in U. S. Patent No. 2,327,066.

In general, the oxygenated group in a product from an olefin by the "oxo" process is thought of as becoming attached to an unsaturated carbon which holds at least one hydrogen atom. In those cases where the carbon monoxide attacks a "saturated" carbon atom, it must be assumed either that that carbon has become unsaturated prior to reaction by a shift of a hydrogen atom or that the attack is directly on a carbon atom that is truly saturated. For example, 2-butene has been reported to give 1-pentanol and 2-methylbutanol in equal quantities, a result that cannot be explained on the simple basis that oxonation takes place only on unsaturated carbons holding hydrogen. 2, 3-dimethyl-2-butene also reacts giving only 3, 4-dimethyl-1-pentanol; in this olefin no hydrogen is attached to the unsaturated carbon atoms and the attack of carbon monoxide must either be on a "rearranged" olefin or on a saturated carbon atom.

It has been found that particularly desirable alcohols for the formation of the formals of this patent can be prepared by the application of the "oxo" synthesis to polymers and copolymers of C_3 and C_4 monoolefins. These monoolefins are readily available in refinery streams and processes for their conversion to liquid copolymers have been worked out by the art. One such process, known as U.O.P. polymerization, consists of passing the olefin-containing stream in liquid phase in contact with an acid catalyst comprising phosphoric acid impregnated on kieselguhr. Other acidic catalysts, such as phosphoric

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acid or copper phosphate impregnated on silica gel, sulfuric acid, Friedel-Crafts catalysts, activated clays, silica-alumina, copper pyrophosphate, etc., may be used. Suitable conditions when employing phosphoric acid catalysts of the U.O.P. type are temperatures of 300° to 500° F., pressures from 250 to 5,000 p.s.i. and feed stocks comprising refinery streams containing propylene and mixed butylenes. Suitable feed stocks, for example, may contain from 15 to 60 mol per cent propylenes, from 0.5 to 15 mol per cent butylenes, and from 0.1 to 10 mol per cent isobutylene, the remaining being saturated hydrocarbons. Other suitable feed stocks are the dimer and trimer of isobutylene.

The preferred "oxo" alcohols employed in forming the formals of this patent are those having from 8 to 20 carbon atoms derived from olefin copolymers having from 7 to 19 carbon atoms. In preparing these oxo alcohols the desired olefin fraction is segregated from the crude olefin polymer product by fractionation.

It will be seen from Table 1 that the oxo formals of the patent compare very favorably with the best synthetic lubricant the art has thus far been able to develop. It is also to be seen that these materials possess viscosity characteristics that make them much more advantageous than mineral oils under conditions of varying temperatures.

It has also been found that lubricating compositions prepared by thickening the formals of this invention, or blends of these formals with other synthetic lubricants, with a grease-forming soap, have desirable properties which make them especially advantageous for the lubrication of moving metal parts where a liquid cannot be used. These grease compositions are simply and economically prepared using, as a thickening agent, any of the common grease forming soaps. The alkali or alkaline earth metal soaps of high molecular weight substantially saturated fatty acids are used and the grease compositions prepared by any of the methods with which the art is familiar. For instance, soaps such as the oleates, stearates or ricinoleates of sodium, potassium, lithium, calcium, barium, strontium, aluminum, iron, zinc, copper and the like may be used as thickeners for the formals of the invention as well as soaps formed by heating animal, fish or vegetable oils with soda, lime, baria hydrate or lithia hydrate. An example of some of these grease compositions is set out below.

EXAMPLE

Formulation:

7.5% lithium stearate
7.5% lithium hydroxy stearate
0.5% phenothiazine
84.5% formal of C_{10} oxo alcohol

Preparation. The combined soaps were dispersed cold in the formal and then heated while stirring to 400° F. At this temperature the inhibitor (phenothizine) was added and the mass poured into a shallow pan for cooling. When cold the grease was homogenized to a soft uniform consistency.

Properties:

Appearance–Light cream color, smooth, buttery.

Penetrations, 77° F., mm./10:
Unworked 280
Worked (60 strokes).....300
Worked (100,000 strokes)...360

Cropping point (°F.).....365

Water solubility Nil(boiling water)
solubility....Nil (boiling water)

Norma Hoffman oxidation
(hours to 5 p.s.i. drop in oxygen pressure) 291

TABLE I
Comparative data for mineral oil and synthetic lubricants

	Mineral Lubricating Oil	*Synthetic Lubricant A*[1]	*Synthetic Lubricant B*[2]	*Formal of C_{10} Oxo Alcohol*	*Formal of C_{13} Oxo Alcohol*	*Formal of C_{16} Oxo Alcohol*
Kinematic Viscosity (Cs. at):						
210° F	7.5	5.1	3.7	2.0	3.4	6.1
100° F	52.7	22.1	15.2	6.4	15.0	46.1
−40° F	Solid	3,900	3,430	693	7,788	.....
−65° F	Solid	Solid	26,000	.....	99,860	.....
Flash Point (°F.)...	445	425	450	345	410	440
Pour Point (°F.)...	−25	−65	<−75	<−75	<−75	−50

[1] 65% di-2-ethylhexyl sebacate+35% of a reaction product of tetraethylene glycol and the half ester of adipic acid with 2-ethylhexanol.

[2] C_{10} Oxo alcohol diester of adipic acid.

Bentonite-Thickened Ester as High Temperature Grease

U. S. Patent 2,761,844 to Gulf Research and Development company. Grease-like lubricants for use in plug valves, for example, at 500°-900° F. are prepared by dispersing 20-40% of finely divided graphite (over 50-200 mesh) and 5-20% bentonite-amine compound (e.g. decylamine bentonite) in an ester base (e.g. di-2-ethylhexyl sebacate).

DETAILS

So far as is known, no satisfactory grease-like lubricants have been developed for lubrication at temperatures up to 900° F. While some lubricants have been suggested for use at these higher temperatures, they have not been entirely satisfactory in that either they are difficult to handle and apply at room temperature or they decompose at elevated temperatures leaving in some instances, resinous deposits, and other instances where complete evaporation occurs, only metal to metal contact. In either of the latter instances seizure of the moving parts results for want of adequate lubrication.

In U. S. Patent 2,761,844 issued

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to Gulf Research and Development company, grease-like lubricants are described which, while useable at below 500° F., are particularly adapted for uses at temperatures in excess of 500° F.

One important use for the compositions is in the lubrication of plug valves. Many of the advantages of plug valves flow from the effective lubricating means provided by their design. Efficient operation of these valves is therefore dependent upon the lubricant employed. Naturally, when these valves are placed in lines carrying materials which have been heated to temperatures of 500° to 900° F., a lubricant is required which will not break down at these high temperatures. If the lubricant breaks down, then frequent valve replacement is required as a result of seizure and galling. Valves which are lubricated with the compositions of this patent can be operated over prolonged periods of time at temperatures of 500° to 900° F.

It has been discovered that lubricating compositions which are easy to handle and apply at room temperature and which give adequate lubrication of moving parts at temperatures of 500° to 900° F., can be prepared by dispersing graphite and a compound of a bentonite and an organic base in an ester of dibasic acid. Thus, it was found that a dispersion of graphite and a compound of a bentonite and an organic base in an ester of a dibasic acid results in a lubricant having good lubricating characteristics at temperatures up to 900°F. The operating life of a valve when lubricated with a composition of the patent is claimed to be increased over the operating life of a valve lubricated with competitive compositions.

The bentonite compounds employed are compounds composed of a montmorillonite mineral in which at least a part of the cation content of the mineral has been replaced by an organic base.

The bentonite-organic base compounds are preferably prepared as described in U. S. Patent No. 2,033,856, by bringing together the bentonite and the organic base in the presence of aqueans mineral acid to effect base exchange. The organic bases should preferably be titratable with mineral acids. Among these reactive bases are many alkaloids, and cyclic, aliphatic and heterocylic amines. Examples of such amines and salts are: decyclamine, dodecyclamine, hexadecycl, ammonium acetate, dimethyldidodecyl ammonium acetate, and the corresponding chlorides and quaternary ammonium chlorides. The organic bases employed should be such as to impart substantial organophilic properties to the resulting compounds. The preferred bentonite compounds are prepared from quaternary ammonium compounds in which the N-substituents are aliphatic groups containing at least one alkyl group with a total of at least 10 to 12 carbon atoms. When aliphatic amines are used they preferably contain an alkyl group with a total of at least 10 to 12 carbon atoms.

Ordinarily the bentonite compound is employed in an amount between 5 and about 20 per cent by weight of the total composition.

The esters employed are those which have a majority of the properties of hydrocarbon oils of lubricating grade such as the lubricating oils customarily used in compounding greases. While esters having viscosities as high as 4000 SUS at 100°F. may be used, it is preferred to use esters having viscosities of about 45 to about 400 SUS at 100°F. If desired, a mixture of esters of suitable viscosity may be employed instead of a single ester, by means of which any desired viscosity within the range of 45 to 4000 SUS at 100°F. may be secured. The viscosity of the ester has little effect on the dropping point of the compositions, but more viscous esters produce compositions having greater stickiness and adhesive properties than do the less viscous esters. Ordinarily the more viscous esters are used in producing lubricants of greater consistency. However, the consistency can also be adjusted by the amount of the bentonite compound used. In addition to possessing lubricating properties, the esters should be substantially neutral. Free acids tend to catalyze the oxidation of the esters, resulting in compounds which are corrosive to metals. Therefore, to reduce a grease-like lubricant which is stable and which is substantially non-corrosive to metals, esters preferably having a neutralization number below about 0.2 are employed.

Some of the preferred esters used in making the compositions are the diesters of aliphatic dibasic acids and alcohols containnig at least four aliphatic carbon atoms, preferably between 6 and 16 carbon atoms.

As examples of some of the dibasic acids suitable for the preparation of the esters used in making the compositions there may be mentioned oxalic, malonic, succinic, isosuccinic, glutaric, ethyl malonic, pyrotartaric, adipic, pimelic, suberic, azelaic, sebacic, and phthalic acid. When a low molecular weight acid is esterfied, a high molecular weight alcohol is preferred in order to produce an ester having a majority of the properties of hydrocarbon oils of lubricating grade. While the diesters of the aliphatic dibasic acids are preferred, the esters of aromatic dibasic acids such as the phthalic acid ester of a material such as castor oil or other high molecular weight alcohols may be used.

The esters may be prepared by any of the known methods. According to one method, as described in U. S. Patent No. 2,091,241, a dicarboxylic acid or its anhydride is dissolved in an inert solvent, after which the resulting mixture is heated to its boiling point. While maintaining the mixture at its boiling point, an alcohol to give the desired ester is added gradually. When addition of the alcohol is completed, the solvent is distilled off and esterification is carried out at a temperature above 150°C.

In most instances, the ester con-

SW's High Efficiency Grease Kettle

This 200 hp Struthers Wells Grease Kettle, shown being checked out at the SW Plant, represents a new design principle which permits heavy horsepower input, counter-rotating de-stratifying arms for "double motion" efficiency, plus hinged scraper blades for ultimate heat transfer. For greater production efficiency for all types of greases, let us design an SW *Double Motion* Kettle to your requirements.

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Offices in Principal Cities

tent comprises about 50 to 70 per cent by weight of the total composition.

The graphite employed is finely divided, i.e., 50 mesh or finer, and advantageously finer than about 200 mesh. The amount of graphite in the compositions can be varied over a wide range without affecting its beneficial effects. However, it is preferred to incorporate graphite in the compositions in an amount between about 20 and about 40 per cent by weight.

In order to illustrate the advantageous properties of the compositions of this patent, a grease-like lubricant was prepared by dispersing 9.8 parts by weight of dimethyldicetyl ammonium bentonite and 33.8 parts by weight of graphite, 98 per cent of which passed a 325-mesh screen, in 56.4 parts by weight of di-2-ethylhexyl sebacate. The di-2-ethylhexyl sebacate had a viscosity of 70 SUS at 100°F., a flash point of 490°F. and a pour point below −60°F. The resulting lubricant had a consistency (ASTM) of 232 unworked and 245 worked. This lubricant was easy to handle at room temperature and was easy to apply by means of a pressure gun through a ball-type fitting to parts requiring lubrication. When this lubricant was applied to plug valves which were in use in a fluid catalytic cracking unit, these valves did not fail even after being operated at 700° to 900°F. for several months. When competitive commercial greases were employed, valve failure occurred after the valve had been in use for only a few hours.

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(See "Pails" in Classified Phone Directories)

Aluminum Soaps

U. S. Patent 2,758,123 to Sun Chemical corporation. Preparation of aluminum soaps (including hexoate) adapted primarily for bodying lighter hydrocarbons.

News Items

New British heat-resistant greases based on molybdenum disulfide and modified bentonite are claimed to give efficient lubrication at 100,000 p.s.i. in machinery operating continuously at up to 450°F., according to Rocol, Ltd., Swillington, Yorks, England (Chemical Week., 2/16/57, p. 96).

Finely powdered titanium disulfide may become a good lubricant, performing better than graphite or molybdenum sulfides, especially at temperatures over 1000°C., according to Australian tests (Chemical Wk., 1/26/57, p. 79).

A new fluorocarbon grease, a blend of low molecular weight polymers of chlorotrifluoroethylene, was introduced by Halocarbon Products corporation, Hackensack, N. J., for use as a lubricant on plug cocks at oxygen plants and in other corrosive applications (Chemical Wk., 1/26/57, p. 62; Chem. & Engrg. News, 1/21/57, p. 80). ■

People in the Industry

Emery Announces Personnel Changes

Joseph P. Clancy has been appointed eastern district sales manager of the organic chemical sales department of Emery Industries, Inc., R. F. Brown, sales manager, announced. Clancy will make his headquarters at Emery's Philadelphia office. He succeeds J. W. Ritz, recently named assistant sales manager.

Clancy Quinty

Brown also announced the following additional changes in the organic chemical sales force: the appointments of Joseph E. Quinty to the Chicago office, William C. Sowers to the New York office, and Arthur R. McDermott succeeding Clancy as New England representative.

Sowers McDermott

Clancy joined Emery in 1949, serving in the southern sales territory. In 1945 he was appointed chemical sales representative in New England. A graduate of Clemson Agricultural college, Clancy is a member of the American Association of Textile Chemists and Colorists, the Boston Rubber Group, the Rhode Island Rubber Group and Alpha Chi Sigma. He also is a director of the eastern New England section of the Society of Plastics Engineers.

Quinty has been with Emery since 1953 in the New York-New Jersey area. A graduate of Purdue university, he is a member of the American Chemical Society, the Society of Plastics Engineers, and the Salesmen's Association of the American Chemical Industry.

Sowers joined Emery in 1953, serving as sales representative in the Cleveland, Detroit and Chicago area. He holds a B.S. degree from West Virginia university.

McDermott has been active in sales and development work since joining Emery in 1956. A graduate of the school of mines and metallurgy of the University of Missouri, he is a member of the American Chemical Society and Alpha Chi Sigma.

General Mills Names Pugh Sales Manager

The special commodities division of General Mills has appointed Fred M. Pugh, of Cleveland, Ohio, as sales manager. Pugh's immediate assignment is supervision of Guar sales. He will later assume sales responsibilities for other products of the division.

Pugh, graduate of Purdue university, has specialized in industrial sales and comes to General Mills with many years' experience in this area. He succeeds W. A. Jordan in sales. Jordan will now devote full time to the research and technical development of Guar gum products.

Kipp Directs Research at Foote Mineral

Dr. E. M. Kipp has joined Foote Mineral company as director of research, L. G. Bliss, president, announced. In his new capacity, Kipp will direct the activities of Foote's research and development department at Berwyn, Pa.

From 1947 until his new association with Foote, Dr. Kipp served as director for one of the research divisions of Aluminum Company of America, having been affiliated with Alcoa in various research capacities since 1939.

Continued on page 46

SWIFT'S INDUSTRIAL OILS INVITE COMPARISON

Write for a trial order of any of these Swift quality products . . . a trial in your own shop will convince you of their stability and dependability in helping to produce lighter and more uniform lubricants.

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Swift

To Serve Your Industry Better

Kipp Directs Foote Research

Continued from page 45

Dr. Kipp received his B.S. in chemistry at Iowa Wesleyan college in 1934; his A.M. degree from Boston university in 1935; and his Ph.D. from Pennsylvania State university in 1939.

He has specialized in physical chemistry and made many investigations in this field. These include: the physical chemistry and metallurgical aspects of metallic interfaces with reference to fundamental properties of friction, wear, and load-bearing capacities; oxidation of hydrocarbons; and power-system oil problems.

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H-27

Hetchler Manages W. G. Smith's New Chemical Division

Werner G. Smith, chairman, announced the appointment of John D. Hetchler to manage the firm's new chemical division, which will be located in Cleveland and will manufacture new products which will expand the company's present line of sperm whale oils, fish oils, and core oils.

Background of Twenty-one Years

A chemical engineer from Michigan State, Hetchler has a background of 21 years in the fats and oils industry. Starting as a chemist with the Wyandotte Oil and Fat company, he served as assistant plant manager and as manager of sales and technical service for the New York area and later as chemical director. Recently, he resigned as sales manager of the chemical products division of Archer-Daniels-Midland company.

Warren's Chief Chemist Post to Meilus

The Warren Refining and Chemical company of Cleveland announced the appointment of Albert Meilus, 33-year-old chemist from Lithuania, to the post of chief chemist in their lubrication division.

A primary duty of the new chief chemist will be to make trips into

the territories to coordinate more effectively the efforts of Warren lubrication engineers in the field with those of the laboratory.

From March 1956 until joining Warren, Meilus was employed in the laboratory of Southwest Grease and Oil company, Wichita, Kansas, developing military and civilian synthetic greases. He also worked on Bentone base greases, a program of consistent improvement of Lithium greases and a cutting oil program.

Escaped from Lithuania in 1944

Meilus and his wife escaped from Lithuania in August, 1944, when his college education was halted by the Russian invasion. He completed his work for a chemical degree at Stuttgart college in the American zone of Germany. Arriving in Canada in 1948, he continued his studies at Toronto University where he received a B.S.A. degree in 1951.

From 1951 to 1956 he worked with the British American Oil company at Clarkston, Ontario. There he spent a year in the refining control laboratory and four years in the technical service laboratory developing new automotive and industrial oils and making detailed analyses of greases and oils.

ASLE Officers and Directors For 1957-58 Announced

The American Society of Lubrication Engineers announced the election of Mr. A. B. Two of the Ford Motor company and Mr. B. T. Harding of the Midwest Oil company to the board of directors of the society. Directors hold office for three years. Mr. L. O. Witzenburg of the Farval corporation was elected industrial director for a term of two years.

Directors elected replace retiring directors A. E. Cichelli, Bethlehem Steel, W. Lasky, Gulf, Mobile & Ohio, and C. R. Schmitt of the E. F. Houghton company, the latter having served as industrial director.

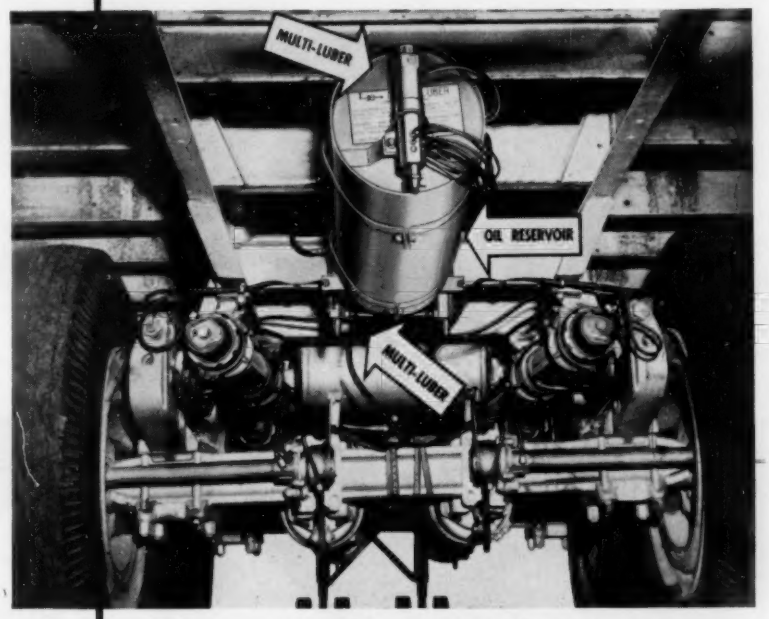
For the 1957-58 year, the following officers of the society have been elected by the board of directors:

Continued on page 48

Lincoln FLEET Multi-Luber*

AUTOMATIC POWER LUBRICATION

Slashes operating and maintenance costs



Air-operated Multi-Luber units mounted on 7-gallon lubricant reservoir, as seen from rear of Freuhauf trailer. Feed lines supply lubricant to axle hanger, bracket assemblies, brake cams and slack adjusters.

Lincoln's Multi-Luber system *automatically* lubricates truck-trailers while they operate . . . doubles or triples the time interval between overhauls . . . and increases net profit per ton mile. Every time brakes are applied, the system cycles, forcing a measured quantity of refinery pure lubricant under high pressure into each bearing.

More than 500 over-the-road fleets are now using Lincoln Multi-Luber. Hundreds of thousands of test miles prove Multi-Luber provides maximum protection against bearing wear . . . smoother vehicle operation . . . elimination of down-time and man-hours for lubrication . . . increased service-life of bearings and moving parts. In fact, the six largest builders of truck-trailers have adopted Lincoln Multi-Luber as a factory-approved, optional accessory!

For complete information, write for Bulletin 532.

Lincoln

THE MOST TRUSTWORTHY NAME IN LUBRICATING EQUIPMENT

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Division of The McNeil Machine & Engineering Co.
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ASLE Officers and Directors

Continued from page 47

President—J. O. McLean, Reynolds Metals

Vice President-at-large—J. D. Lykins, Wheeling Steel

Secretary—J. L. Finkelmann, Cycleweld Div., Chrysler

Treasurer—J. W. Peterson, Standard Oil (Indiana)

Eastern Regional Vice Pres.—M. E. Dougherty, Acheson Colloids

Mid-Western Regional Vice Pres.—A. B. Wilder, du Pont

Western Regional Vice Pres.—R. C. Dishington, General Petroleum

Officers are elected for a one-year term and the vice president-at-large succeeds the president in that office.

To fill the unexpired term of office for director, created upon the election of Mr. Lykins to vice president-at-large, the board of directors elected Mr. S. R. Calish, Jr., of the California Research corporation for a one-year term.

Moore Will Service Lithium's Midwest Sales

J. D. Campbell, vice president, sales, Lithium Corporation of America, Inc., announces the appointment of M. Malcolm Moore as the company's representative in charge of the midwest sales area with offices located at 121 West Wacker drive, Chicago. Prior to joining Lithium Corp., Moore was branch manager of Pure Oil company at Flint, Michigan. He is a graduate of Williams college(B.A.) and Harvard Business school (M.B.A.)

Nopco Chemical Opens Los Angeles Sales Office

J. E. Connell, sales manager of the West Coast division of the Nopco Chemical company, Richmond, Calif., has announced the opening of a Los Angeles sales office for Nopco's industrial chemicals division and the Metasap Chemical company, Nopco's wholly-owned subsidiary.

Working out of the new office, located at 4858 Valley Boulevard, will be F. R. Gagnier and D. E. Murphy who will handle the sales of Nopco's many industrial processing chemicals.

The new office is the result of Nopco's efforts to maintain a close working relationship with its customers. Telephone communications with Nopco can now be completed by calling CApitol 1-3128.

News continued on page 50

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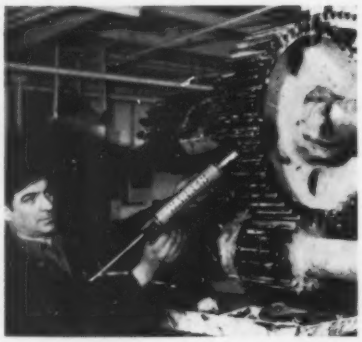
Industry News

Warco Coupling Lubricant for All Threaded Assemblies Just Developed by Warren

Development of lubrication for all threaded assemblies has just been announced by the Warren Refining and Chemical company of Cleveland.

Because of its non-melting and extremely high adhesive and load carrying characteristics, Warco Coupling Lubricant, the new development, provides maximum lubrication for threaded assemblies where high pressure, shock loading, and high temperature conditions prevail.

Among other applications, it is recommended for drill collars, tool joints, drill tubing, drill couplings, and tool bit threads.



For high pressure, shock loading, and high temperature

In addition to the characteristics already mentioned, Warco Coupling Lubricant has the following valuable properties: easy break-out; no galling or seizing; inseparability; inability to dry out; pumpability at sub-zero temperatures; unvarying elasticity under all work conditions; high fluidity; imperviousness to water and no known melting point.

Warco Coupling Lubricant should find immediate acceptance by quarries, mines, contractors, and all other users of drill equipment.

Kerr-McGee Contracts With C. W. Nofsinger And Refinery

A contract for the design and construction of new facilities for the Wynnewood, Oklahoma, refinery of Kerr-McGee Oil Industries, Inc., has been awarded to the Refinery Engineering company of Tulsa. Engineer-architect on the project is the C. W. Nofsinger company of Kansas City, Missouri.

Two unifiners and the platformer are being built under license from Universal Oil Products company, Des Plaines, Illinois.

Kerr McGee Acquires Triangle Stock

Also, all of the capital stock of Triangle Refineries, Inc., of Houston is being acquired by Kerr-McGee, according to an announcement by Dean A. McGee, Kerr-McGee president, and J. B. Saunders, Triangle president.

Will Retain Triangle Name

Triangle will continue to operate under its same name, as a subsidiary of Kerr-McGee, with the same management and personnel as before. J. B. Saunders, founder of Triangle, will continue as president and chief executive officer. The other officers of Triangle all of whom will be retained in their present capacities are: J. Howard Barksdale, executive vice president, Hubert H. Raborn, vice president and secretary, and C. D. Tinsley, vice president. The company has some 220 employees.

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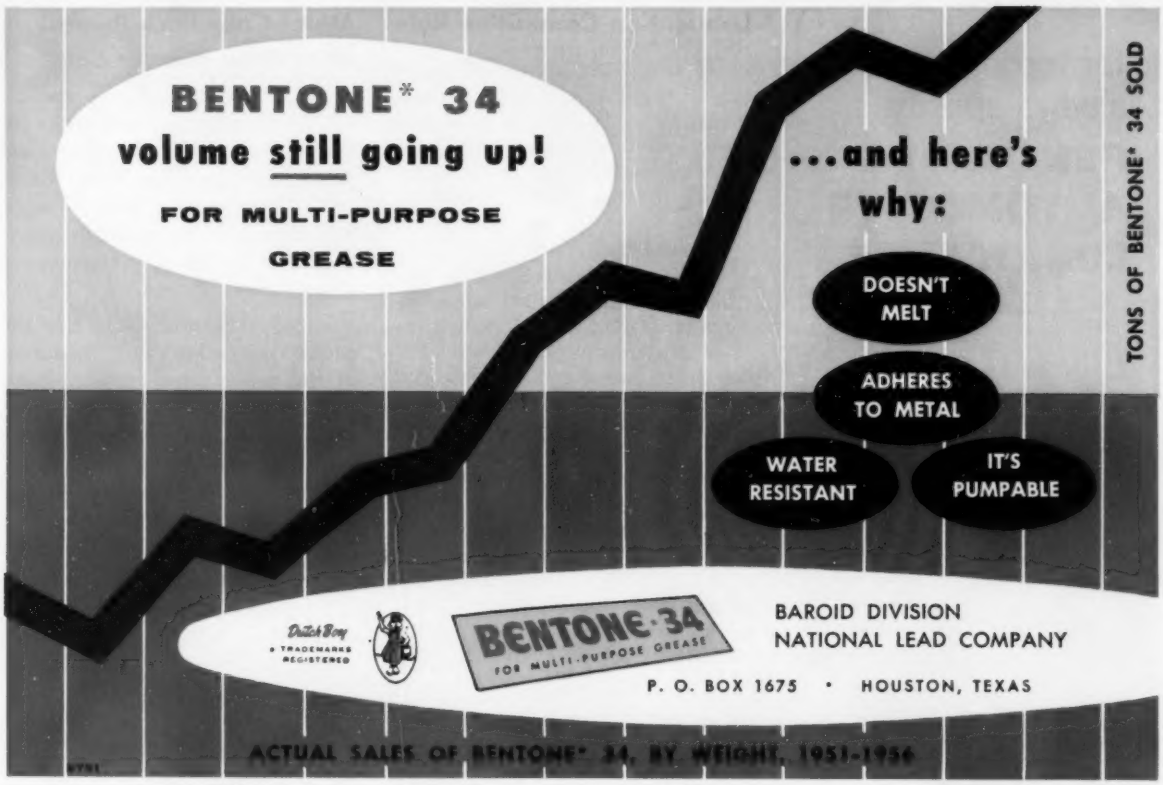


Gulf's D. P. Clark Discusses Role of Lubrication Committee

The role played by the oil industry in providing lubrication services to the consumer was the keynote of an address made May 6 at Point Clear, Alabama, by D. P. Clark of Gulf Oil corporation.

Speaking to the Lubrication Committee of the American Petroleum Institute gathered for its semi-annual meeting, Mr. Clark, one of the organizers of the 24-year-old group and an NLGI Board member, re-affirmed some of the Committee's original objectives.

It was the aim of its originators, reminded Mr. Clark, for the Lubricating Committee to act as a clearing house for technical problems pertaining to the use of lubricants and proper selection for the purposes intended and their correct application.

As an objective, the Committee was to correlate and disburse technical data on lubricants and devices. Such information, when properly applied by the consumer, could assure him of better service, increased production, and lower maintenance costs for his machines as well as avoidance of plant closedowns due to faulty lubrication.

World War II Contribution

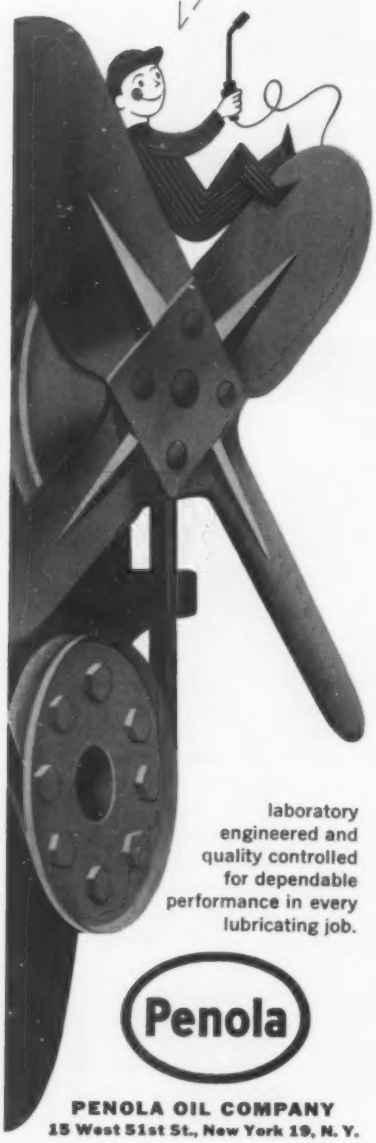
In tracing the history of the Lubricating Committee's success in realizing its goals and its continuing awareness of its objectives today, Mr. Clark stressed the value of the counsel given by oil company lubrication engineers to industrial plants.

Such service often goes beyond the ascertainment of requirements and recommendations of lubricants' use and suitability for the consumer's need. Mr. Clark cited illustrations where lubricating engineers actually have shown industries how to improve production particularly in connection with services performed at the instigation of the Lubricating Committee as part of its contribution to the defense effort during World War II.

In addition, declared Mr. Clark, the Lubricating Committee has devoted time to a number of discussions with manufacturers' engineers, concerning machine engine design problems. And the Committee's activities have not stopped there. The Gulf Oil official said

Continued on page 52

for top protection,
always specify
PENOLA AUTOMOTIVE LUBRICANTS



laboratory engineered and quality controlled for dependable performance in every lubricating job.

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Lubrication Committee Role

Continued from page 51

many times discussions are desirable in the field of management in order to make certain that the brain child of the engineer or his lubrication recommendations are proper for the greatest consumer benefit.

Individual "Price Tags"

Mr. Clark also declared the petroleum industry today has lubricants to market in which each individual company puts to work the best manufacturing and research techniques available to them.

"We must and should combine the merchandising of these products with the best engineering technical field skill that we have available. Combining these into a whole package, these are presented to the consumer. The price tag placed upon this combined product and service is the amount which we each individually happen to believe the package is worth."

Shell Oil Receives Defense Reserve Award

Shell Oil company has received the Department of Defense Reserve Award for "outstanding cooperation" to the reservists and reserve activities.

The award, consisting of a pennant and a certificate of citation, was made during ceremonies held at the Waldorf Astoria.

Making the presentation in the name of Secretary of Defense Charles E. Wilson, was Rear Admiral Milton E. Miles, Commandant, Third Naval District. Dr. M. P. L. Love, Shell's manufacturing vice president, accepted the award as the representative of H. S. M. Burns, the company president. The ceremonies were attended by executives of Shell and high ranking military officers representing all branches of the armed forces.

In making the award, Admiral Miles said Shell has frequently given assistance and advice to the armed forces in petroleum research. He also pointed out that Shell makes company facilities available for naval reserve use.

Metal Cans Pack Power With New Booster Shot

Metal cans are boosting the use of "Booster Shot," a new super lubricant for outboard motors and power mowers. The Pyroil Company, Inc. of La Crosse, Wis., is now packing this product in American Can company's three-ounce round (202x214) can.

Pyroil "Booster Shot," the packer said, allows full utilization of the engine's horsepower, keeps plugs clean and reduces friction. It also increases gas mileage. Laboratory tests have proved that this lubricant reduces wear by 40 per cent, the packer stated.

The new product retails for approximately 35 cents a can or three for a dollar. It will have national distribution through automotive jobbers and will be sold in sporting goods, hardware and chain stores.

Rapeseed Oil Greases Now Available

Samples of refined rapeseed oil are now available from George Degen and Company . . . considerable research is being devoted to the possible use of this product in multi-purpose greases. The firm writes:

"Greases made from lithium as well as lithium/calcium soaps of hydrogenated rapeseed oil and napthenic mineral oil appear to give

Continued on page 54

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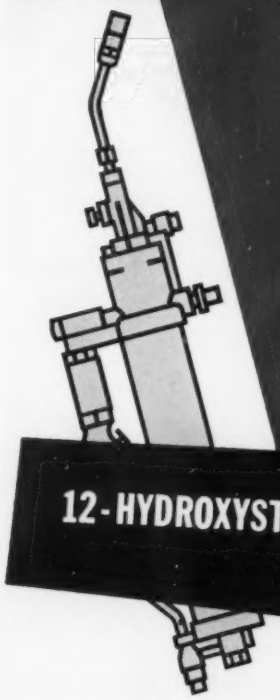

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Acid Value	2.	178.	4.
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Rapeseed Oil Greases

Continued from page 52

considerable promise as substitutes for lithium/calcium 12 hydroxystearic acid greases. While the work stability characteristic currently requires improvement, the water resistance appears better, the drop point slightly lower but still amply high for automotive service, with the yield slightly lower than with lithium stearic acid greases. The work stability is similar to the lithium stearic acid greases."

Barium/rapeseed oil greases are currently under investigation but it will probably be several months before Degen will have any information available. Limited samples are now being produced on a laboratory basis of hydrogenated rapeseed oil . . . these too can be obtained by writing George Degen & Company, 111 Broadway, New York 6.

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Stauffer Offers New Research Chemical

Stauffer Chemical company is now offering a new organic sulfur derivative, Trichloromethanesulfonyl Chloride, in research quantities.

The compound is a white solid, stable at normal temperatures but readily sublimed. It is soluble in a broad range of organic solvents.

Preliminary investigations indicate that the chemical will find major use as an intermediate for the manufacture of pharmaceuticals, dyestuffs, rubber chemicals, and lube additives.

Trichloromethanesulfonyl Chloride is reported to accelerate ignition and improve the lubricating qualities of diesel oil fuels, and to be a vulcanizing agent for GR-S rubber and for natural rubber in the presence of elemental sulfur.

Because of its reactive nature, it is anticipated that the chloride will hold promise as an intermediate in many fields of synthesis.

Emersol Stearic Acids Booklet Available

Emersol Stearic Acids is the title of a new, 24-page booklet offered by Emery Industries, Inc.

Available upon request, the brochure is designed to aid in the selection of the proper grade of stearic acid for each end use. It interprets the results of analytical tests commonly performed on stearic acids, and contains a discussion of physical properties of commercial stearic acids and their relation to its palmitic stearic acid ratio. Tables are given listing the composition of typical grades of stearic acid, as well as a stearic acid selection chart.

Also included in the booklet are a selection covering the handling of stearic acid, and a complete description of all grades of Emersol stearic acids, with emphasis on the significance of their outstanding oxidation and color stability.

Emersol Stearic Acid can be obtained by writing: Emery Industries, Inc., Dept. 5, Carew Tower, Cincinnati 2, Ohio.

API Quarterly Edited by Mellinger

Appointment of Edward D. Mellinger as editor of the American Petroleum Institute *Quarterly* was announced by H. B. Miller, director of the Department of Information. Mellinger, who had been serving as consulting editor, succeeds Walter Henry Nelson, who has resigned. Mellinger has been with the Institute since 1948.

American Potash Expands West Coast District Offices

American Potash & Chemical corporation has announced an expansion of its San Francisco district office, according to William M. Clines, western general sales manager.

E. Robert Little, Jr., has joined

Continued on page 56

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American Potash Expands West Coast District Offices

Continued from page 54

the company as area sales representative under the direction of Rod Taft, district manager.

Clines said the expansion was due to a rise in markets for both industrial chemicals and agricultural chemicals produced by the company.

Little was with Olin Mathieson before joining AP&CC. Previously he was with Holly Sugar and Agriform company. Born at Peabody, Mass., Little received his bachelor of science degree in agriculture at Davis, Calif., campus of the University of California.

Rice Named Chief Chemist Of AP&CC Nevada Plant

Robert P. Rice has joined American Potash & Chemical corporation as chief control chemist at the company's Henderson, Nev., plant, according to an announcement by John S. Vander Laan, AP&CC administrative assistant at Henderson.

Rice formerly was chief chemist at the Jersey City, N.J., plant of Columbia Southern Chemical corporation. He graduated from Siena college at Londonville, N.Y., and took post-graduate work at Rensselaer Polytechnic at Troy, N.Y.

Vacation Schedules For Nopco and Metasap Announced

The annual two-week vacation shutdown of plants of the industrial manufacturing division of the Nopco Chemical company and its subsidiary, the Metasap Chemical company has been announced.

Shutdown of the Harrison, New Jersey plant will be from Friday, June 28 to Monday, July 15. The Cedartown plant will be closed from Friday, August 9 to Monday, August 26.

Nopco and Metasap customers are urged to place their orders well in advance to avoid delays in shipping during the vacation periods.

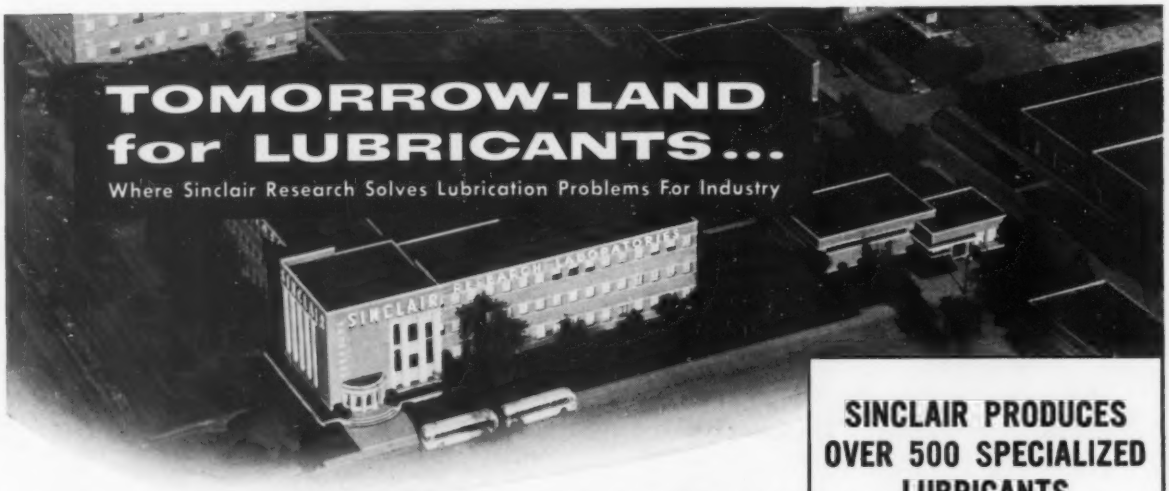
Marietta College Receives Continental Oil Scholarship

Marietta college's Edwy R. Brown petroleum department has announced approval of an annual scholarship program with the Continental Oil company.

The college will receive $1000 annually, half of which is to be awarded to a senior candidate for a degree in petroleum engineering, and half to the college's petroleum department.

The $500 scholarship award is to be presented to a qualified student selected by the petroleum department and is to be used for furthering the student's studies during his final academic year.

A supplementary $500 grant is to go directly to the college for use in the petroleum department.



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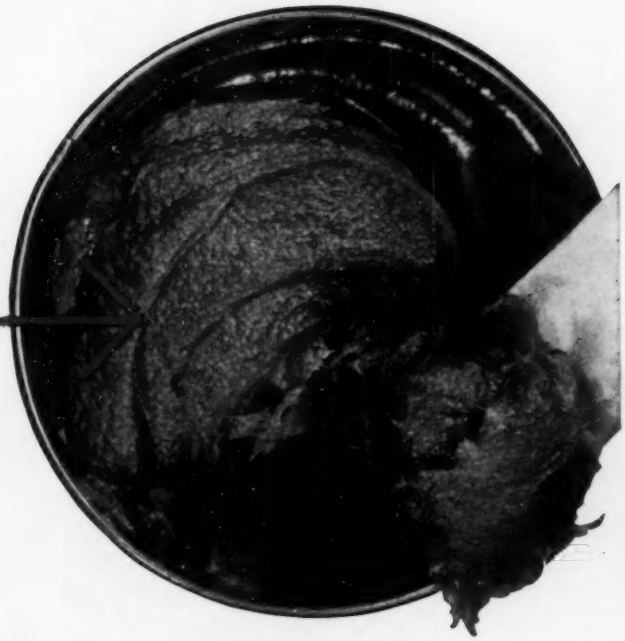

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American Lithium Institute Joins NLGI

New Member

THE AMERICAN LITHIUM Institute was formed in November, 1956, by the three major producers of lithium and lithium compounds to fill the need for a central organization which would serve as a clearing house for technical information on these materials and direct research on problems of an industry-wide nature.

Aware of the rapid expansion of industrial uses for lithium in recent years, the founding companies—American Potash & Chemical corporation, Foote Mineral company and Lithium Corporation of America—decided that the still unexplored potential of this unique metal and its derivatives could best be realized through joint action. By establishing the Institute, they have provided a means for securing fundamental data needed to advance new and promising applications for lithium, and for collecting and disseminating information that will increase lithium's value in both new and established fields of use.

The new Institute, which has its offices in Princeton, New Jersey, will support an extensive program for research and development on problems involving lithium use in such fields as organic and inorganic chemistry, electrochemistry, ceramics, metallurgy, nucleonics and catalytic processes. Projects, selected with the guidance of the Institute technical policy committee composed of member company representatives, will be assigned to appropriate colleges and research institutions for their staffs to carry out in their laboratories.

The information center is located at the Institute offices where a full-time librarian is in the process of setting up a reference library under the direction of Marshall Sittig, Institute president and managing director. Data on all aspects of lithium technology will be gathered for correlation and dissemination to technical people in various consuming industries. In addition to answering specific questions, the Institute will prepare booklets and articles on lithium and its compounds, and will issue a periodic bulletin on new lithium applications. A major project already under way is an American Chemical Society monograph on lithium and its compounds.

Of the established applications for lithium expected to be expanded through Institute activity, the one probably most familiar to NLGI members is the use of lithium soaps in multi-purpose greases and lubricants. One of the largest volume uses for lithium, it is based on the water insolubility, gel-forming properties, good lubricity and high melting points of lithium soaps such as lithium stearate. Added to greases, lithium soaps yield products which have high melting points; are soft enough to flow and lubricate at below-zero temperatures; do not emulsify with water; and exhibit a minimum change in consistency with changes in temperature. Because of this combination of properties, obtainable only with lithium soaps, manufacturers can produce multi-purpose greases and lubricants which are effective over a wide range of use conditions.

Since lithium soaps possess good lubricating properties themselves, they are also being used as lubricants in powder metallurgy. Mixed with the powdered metal prior to pressing, they serve as internal lubricants and as die lubricants, giving denser compacts with low mold-release pressure and low die wear.

Company and Technical Representative

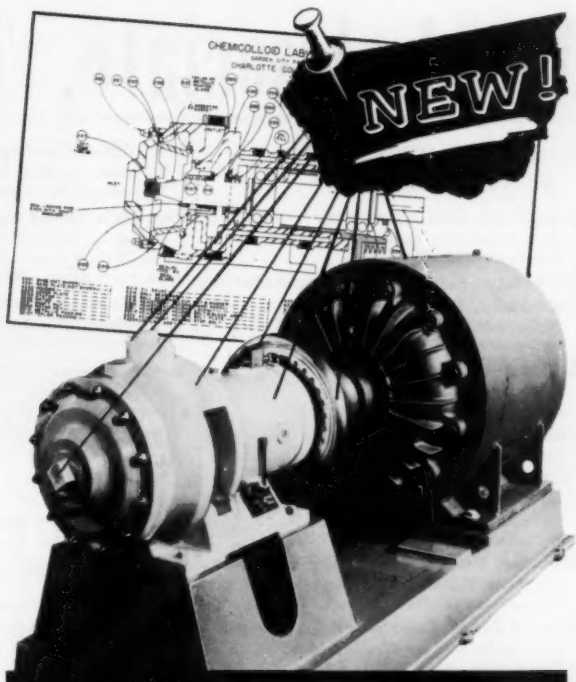
Sittig

Marshall Sittig, American Lithium Institute's NLGI representative and technical committee member, brings to NLGI varied professional and business experience as a chemical engineer in research, production, technical advertising and technical writing. An authority on the alkali metals, he is widely known for his writing on products and processes of the chemical and petroleum industries.

After graduating from Purdue university in 1940 with a chemical engineering degree, Mr. Sittig served a short stint as a process design engineer for Standard Oil of California. He left that company in 1941 to pursue graduate work in chemistry at Purdue, instructing in chemistry as well. Later in the same year, he took a position with E. I. du Pont de Nemours & Co. where for five years he was a production supervisor in the neoprene synthetic rubber plant, and later was assigned to chemical engineering research on new chemical reactions at high pressures. Joining the Eastern corporation in 1948, he conducted chemical engineering studies related to paper manufacture. While with Eastern Mr. Sittig published the first of many technical articles.

In 1949, he became interested in technical communications as a career and he accepted a position with the Chrysler corporation in literature searching and technical report writing and editing. He left Chrysler in 1951 to do technical writing and editing at Ethyl corporation, where, specializing in the dissemination of technical information on sodium, he became manager of technical advertising. Mr. Sittig's writing has been so prolific that in the last seven years he has written over forty technical articles, two chapters for technical books and one book, *Sodium—Its Manufacture, Properties and Uses*, which has just come off the press.

With the formulation of the American Lithium Institute, Inc., in November, 1956, Mr. Sittig was elected president and managing director by the member companies. In his new capacity, he is directing the Institute's programs of research on industry-wide problems and dissemination of technical information on lithium and its compounds. His office is at Institute headquarters in Princeton, New Jersey.



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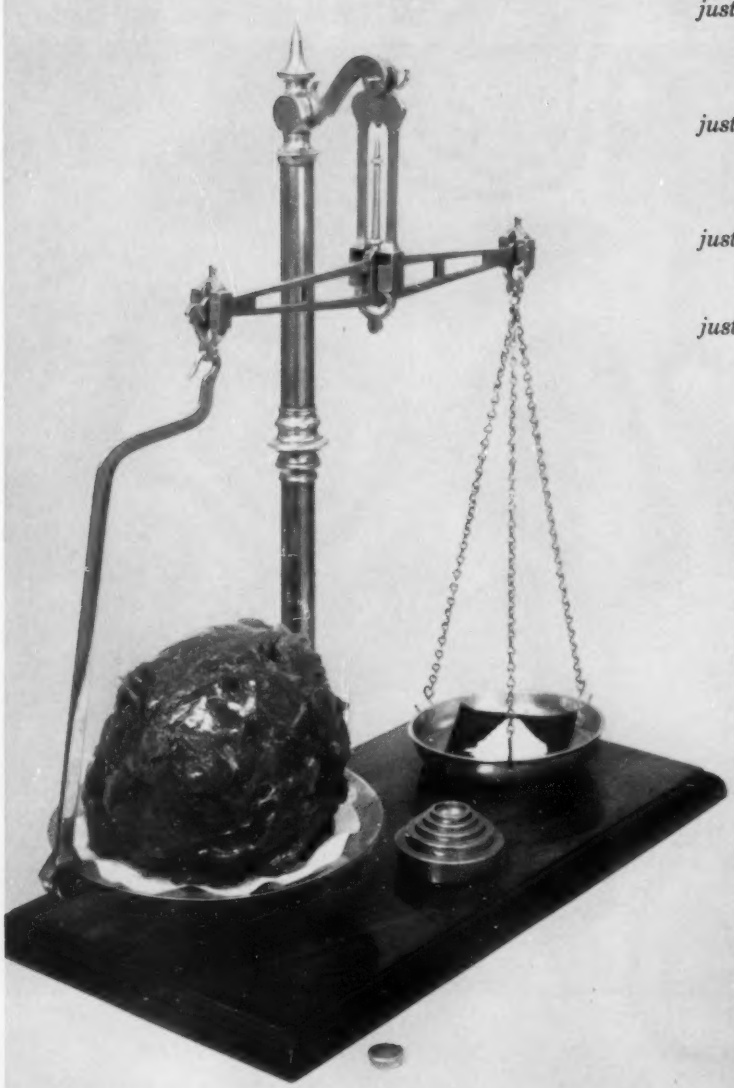
Representative—Richard Bender

Petroleum Educational Institute
9020 Melrose Avenue, Los Angeles 46, Calif.
Representative—G. A. Zamboni

Phoenix Chemical Laboratory, Inc.
3953 W. Shakespeare Ave., Chicago 47, Ill.
Representative—Mrs. G. A. Krawetz

Products Development Laboratory
1 Market St., West Warwick, Rhode Island
Representative—Alberic T. DiMasi

THE THINGS YOU CAN DO WITH A LITTLE LITHIUM



just one pound of lithium hydroxide is all that's needed to make one hundred pounds of multi-purpose grease.

just one pound of finely divided metallic lithium is catalyst enough for one thousand pounds of synthetic rubber.

just one pound of metallic lithium degasifies one thousand pounds of molten copper.

just one pound of lithium hydride releases fifty cubic feet of hydrogen.

It's not so much *the things you can do with lithium* . . . as it is *how little lithium you need to do them*. Present day industrial applications require such small quantities of the metal that today's proven reserves are more than adequate to take care of all foreseeable needs. And even if lithium uses expand faster than their present amazing rate, it's reassuring to note that Foote Mineral, with the largest operating deposits in the United States, is still proving new reserves every day.

If you're interested in how a little of the right lithium chemical might go a long way in improving your operations, we'll be happy to discuss lithium chemistry with you at your convenience. Or, write now for a brief working rundown in "The Physical and Chemical Properties of Lithium Compounds" to Foote Mineral Company, 460 Eighteen West Chelten Building, Philadelphia 44, Penna.

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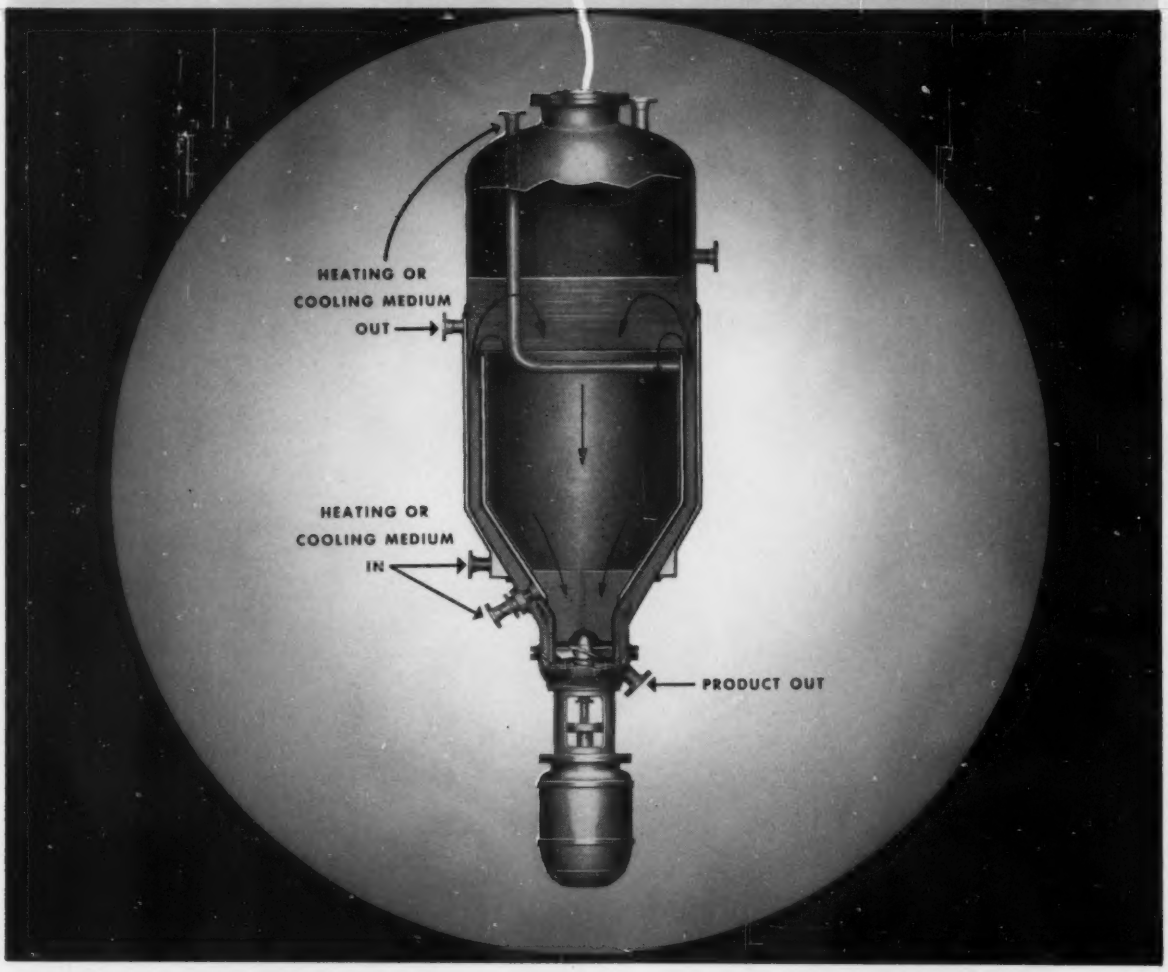

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